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STUDIES ON INDUSTRIAL COMPOUND TECHNOLOGY OF RIGID AND PLASTICISED PVC

A MAJOR PROJECT REPORT SUBMITTED TOWARDS THE PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF THE DEGREE OF

MASTER OF ENGINNERING
IN
POLYMER TECHNOLOGY

By
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CERTIFICATE

*This is to certify that **Mr. Vinod Kumar Bhati** M.E. (Polymer Technology) Student. Delhi College of Engineering has satisfactorily completed his major project entitled **"Studies on Industrial Compound Technology of Rigid & Plasticised PVC"** in partial fulfillment for the award of the Master of Engg. in Polymer Technology of University of Delhi. This thesis is an original work which has not been submitted to any other institute, University or organization for the award of any degree or diploma to the best of our knowledge.*

I wish him all the best success in life

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TO WHOM SOEVER IT MAY CONCERN

This is to certify that Mr. Vinod Kumar Bhati S/o of Shr. Prem Nath Singh, a student of M.E.. (Polymer Technology) of Delhi College of Engineering, Delhi has undergone in Plant Training with us for a period of 6 months commencing from 15.06.99 to 06.12.99.

During this period of training, he was exposed to major project work and other related field of R & D activities..

He was found sincere, punctual and will behaved with his seniors and colleagues during the above period

We wish him all success in every sphere of his life

For PREMIER POLYFILM LIMITED,

(R.P. JAIN
GENERAL MANAGER (P&A)

ACKNOWLEDGEMENT

I wish to express my profound sense of gratitude and indebtedness and heartfelt thanks to my esteemed supervisor, **Dr. A.P. Gupta**, Prof in Polymer Technology, Department of Applied Chemistry, Delhi College of Engineering for his inspiring and invaluable guidance, deep interest and continuous encouragement during the entire course of Project Work The constructive suggestions and enlightening views given by him have been of paramount importance in articulating the whole work

Respectful acknowledgement's made of Prof G L Verma, head of Department of Applied Chemistry, Delhi College of Engineering, Delhi for his valuable suggestions and constant encouragement during the course of this study

I am indebted to Dr Sanjeev Kumar Verma who have gone out of their way to help me to their expertise experience to encourage me with love and affection and I am also thankful to my friend Mr B B Sharma, Avdesh Kumar Sharma

At last, but definitely not least I appreciate the high value of love and encouragement of my parent Shri Prem Raj Singh, Principal, H L M Inter College, without which this undertaking would have been impossible I owe many thanks to them

(VINOD KUMAR BHATI)

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Industrial Compound Technology of Rigid and Plasticised PVC

INTRODUCTION

The compounding process represents the link between raw material production and finished-article manufacture. Its function is to combine the PVC resin with the various additives required for processing and for the service additives properties of the final product, in accordance with the formulation.

There are five general types of industrial PVC compounding operation: -

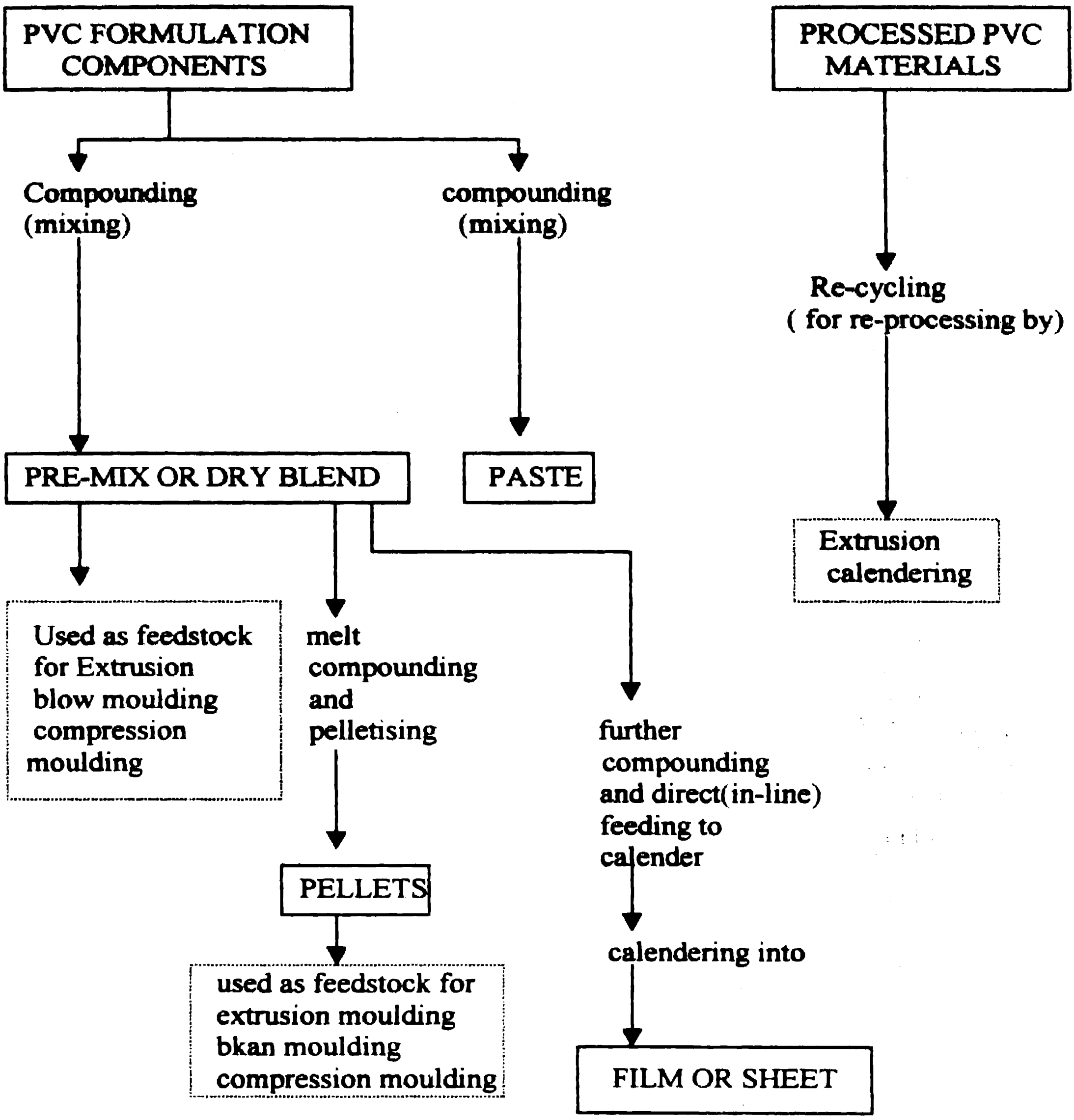
---- preparation of pre-mixes and dry blends.
---- melt compounding and pelletising.
---- compounding for the feeding of film and sheet calenders.
---- production of pastes (plastisols, organosols, plastigels).
---- recycling.

The equipment required can be divided into the upstream section ahead of the compounder, the compounder itself, and the downstream equipment. The upstream units are more or less identical for all the five general types of compounding operation, but the compounder and its downstream equipment have to be adapted to the specific requirements of each type.

The upstream equipment handles the raw materials: it comprises silo storage, conveying, weighing. Included in the compounding section is the PVC pre-mixing operation. The actual compounding and, where pellets are produced, the pelletising operation.

The proportions produced, respectively. from Pre-mix and from pellets as the feedstock. To interpret the table properly. One should bear in mind that the production of film, sheet and board, and of products from plastisols, involves processes with an in line compounding step between pre-mix and final product. Thus, in these cases final-product processing follows directly on the compounding operation, and there is no need either for pelletising or for the downstream equipment that normally follows that operation.

The downstream equipment normally employed for pellets and dry blends handles the cooling, conveying, storage and packaging of the compound.

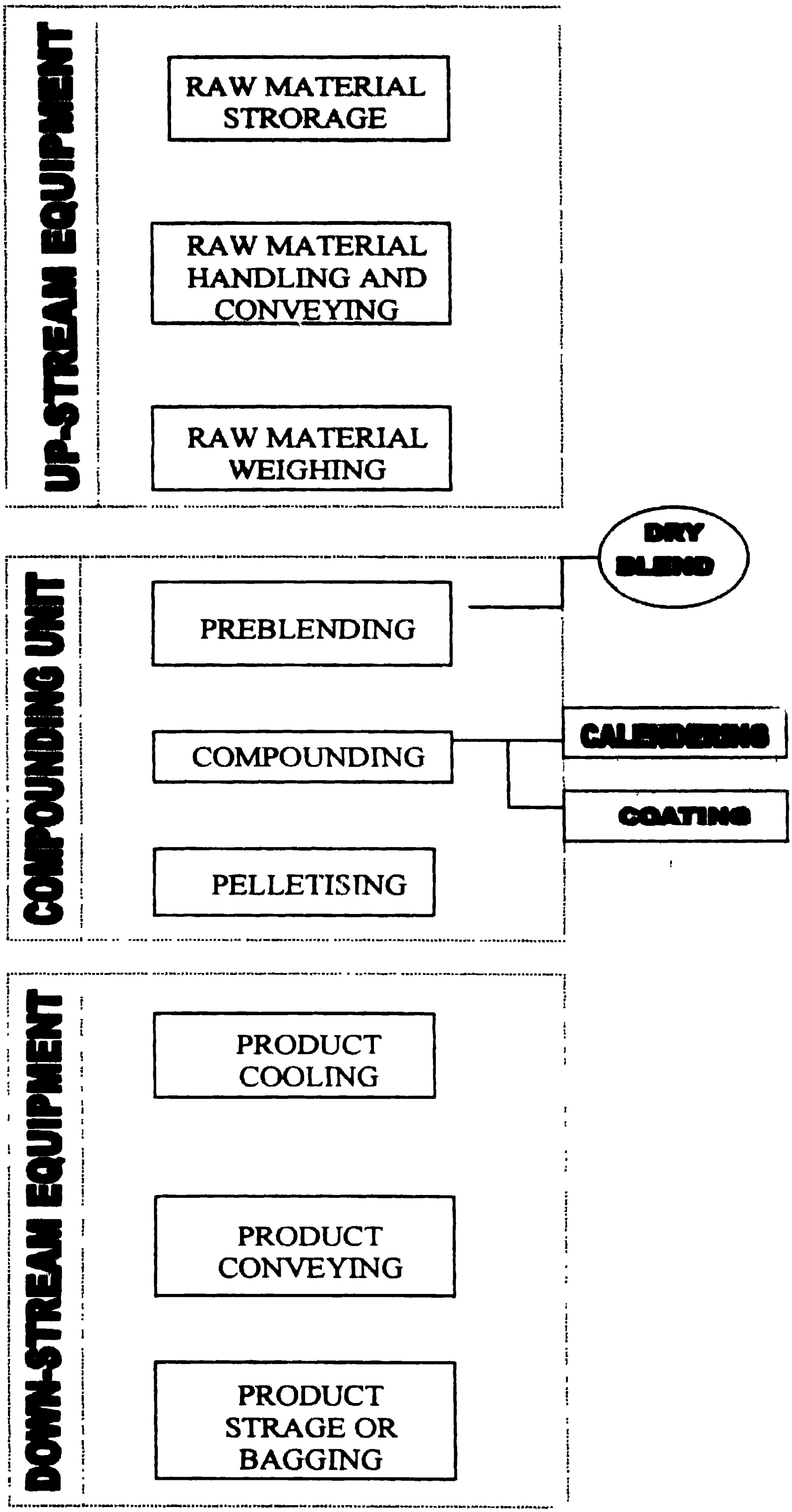

PVC FORMULATION COMPONENTS
Compounding (mixing)
compounding (mixing)
PRE-MIX OR DRY BLEND
PASTE
Used as feedstock for Extrusion blow moulding compression moulding
melt compounding and pelletising
PELLETS
used as feedstock for extrusion moulding bkan moulding compression moulding
further compounding and direct(in-line) feeding to calender
calendering into
FILM OR SHEET
PROCESSED PVC MATERIALS
Re-cycling (for re-processing by)
Extrusion calendering

Industrial compounding of PVC : general schematic outline.

RAW MATERIALS

PVC Polymer and Fillers

In terms of the amounts used in PVC compounding . these are two principal solid raw material components : both are in power from.



PVC compounding : block diagram

From the standpoint of the compounding or extrusion operation, it is important to emphasise those properties that are crucial to the production of free-flowing, dry powder blends.

proportions of Important PVC Product, Respectively, from Pre-mix and Pellctised compound (word-wide)

Products	Produced from :pre-mix(%)	pellets (%)
Extrusions (pipes, profiles, tubes, hoses. siding)	90	10
Injection mouldings	25	75
Cables	—	100
Records	75	25
Blow moulding (bottles)	70	30
Film and sheet	100	—
Plastisol product	100	—

Some properties of PVC Polymers

Properties	Polymer type					
	M-PVC		S-PVC	E-PVC		
K value (DIN 53 726)	57- 71		55-71		65-75	
Processing	---		—	Dried with rolls	Sprayed	Sprayed by special process
Particle shape	Riven porous	Riven Porous	Bead-shaped, compact glassy	Flaky	Spherical	whole and broken spheres
Particle shape	up to 1000	60-250	60-250	60-500	up to 60	up to 200
Bulk density (g^{-1})	540-630	400-620	600-700	300	400-600	300-600

(DIN 53 468)						
Free-flowing properties	Good	Good	Good	Good	Poor	Good
Plasticiser absorption (ml DOP per 5g PVC)	1.5-5	5	2	3-5	0.5-1	3-5

Fillers ·

The effect of fillers on the production of hot blends depends on the loading, particle size and plasticiser absorption. High loading of fine grained filler make blends flow less freely. filler with a porous surface often absorb too much plasticiser, which in turn result in dry mixes. One positive effect worth mentioning use of very small amounts of colloidal silica for powering poorly flowing blends in the cold mixer.

Plasticisers

Plasticisers are the principal liquid components employed in PVC compounding.

Other Additives

The other constituents of PVC compositions, which--from the point of view of compounding --may be regarded as additives to the PVC polymer,

UPSTREAM EQUIPMENT (SILO STORAGE TO WEIGHING)

Silo Storage of PVC Polymer and Fillers

Storage of the solid raw material components calls for buildings designed to house bagged or container goods, or for silo installations. For economic reasons, preference is normally given today to batteries of silo capable of holding several thousand tonnes.

(a) Silo Sizes

Silos with capacities of 150 to 250 m^3 are general used for storing PVC in the plastics industry. Whilst smaller silos are also used , those with volumes of less then $50m^3$ are regarded as uneconomical. This minimum size is set by the capacity of the rail tank cars normally employed now a days for delivering the raw materials from

production plants. The ability to discharge the entire contents of a tank car into an empty silo in a single operation is essential. Otherwise, unnecessary waiting time would result for the tank cars.

On the other hand, the maximum silo size depends on the transport possibilities from the silo fabrication plant to the erection site if finished silos have to be transported by road or rail, the acceptable volume is limited to $150m^3$. Typically, the design diameter of the silo tank is 2.4m, though 3.5m silos are built occasionally. larger units-- up to a volume of $400\ m^3$ for PVC -- can only be transported by water or , if this is impossible, shipped in pieces and welded together on site.

In the case of fillers silos of volume greater then $150\ m^3$ are hardly ever employed because of the relatively high bulk density of the contents.

The size of a battery of silos in a plastics plant depends on the procurement possibilities for the raw materials, raw material consumption, the plant's geographic location, and not least the market situation in the raw material sector.

(b) Material of Silo Construction

Nearly all the silos erected out - of - doors today are fabricated from an aluminium/ magnesium alloy (AlMg3). it is fair to say that the steel silo with internal coating and external paint finish has been displaced by the standard aluminium alloy silo in the field of PVC compounding. Aluminium alloys are weatherproof, require no maintenance (as on paints peels off and on rust develops) and have a virtually unlimited service life. The plates used have a smooth surface, with a peak-to-valley depth normally less than 20 um. Silo walls of aluminium alloy are much less prone to adhesion of contents than those of other materials. There are on problems with electrostatic charges, because unpainted aluminium is an excellent conductor of electricity. Because the external wall reflects well, there is little product heating as a result of exposure to sun light.

(c) Raw Material Intake (silo filling)

With increasing use of silo storage facilities, the traditional practice of purchasing solid raw material components in bags or other small containers is a being increasingly replaced by bulk purchase with delivery by tanker transport. The advantages are :

---- less labour ;
---- no lose of material in transport ;
---- lower raw material prices;
---- no contamination of the material and dust free working condition.

The tilling of raw material silo is always accomlished with pneumatic conveying systems. Both road tankers and rail conveying pressures. Compressed air, and not a suction system, is normally used to empty the vehicle tanks. Screw compressors are used ton generate the required flow of oil-free conveying air. Though most road

tankers have their own compressors, a stationary compressor installation is required at the plant for emptying rail tank cars.

Generally, each silo has its own pipe leading from the connection point for the filling hose.

In order to retain flexibility with regard to raw material, and the ability to handle small batches of different formulation and qualities smoothly, most plant have an additional dumping station for filling the storage silo (or holding bins, or both) with bag goods. Because the amount of bagged goods is usually very small compared with total plant through put, a manual dumping station is normally sufficient for this purpose. The hourly filling rate achievable by manual opening and dumping of bags is about 3 tonnes. To eliminate bag scrap, it is general advisable to follow the dumping station with a suitably dimensioned sifting machine before the raw material is conveyed pneumatically to the storage silo through a rotary valve or a pressurised tank. The bag-dumping station must be arranged so that, instead of escaping. The dust raised during dumping is drawn off to a filter by a suitable exhaust system. As a rule these bag-dumping station are supplied with a built-on filter, so that the filter can be cleaned mechanically after each filling operation to return the dust to the raw material.

A proper exhaust system for a bag-dumping station should be laid for an air flow of about 20 m^3 min^{-1} at a vacuum of mm w.g. The resulting air withdrawal velocity during dumping is about 0.5 ms $-^1$

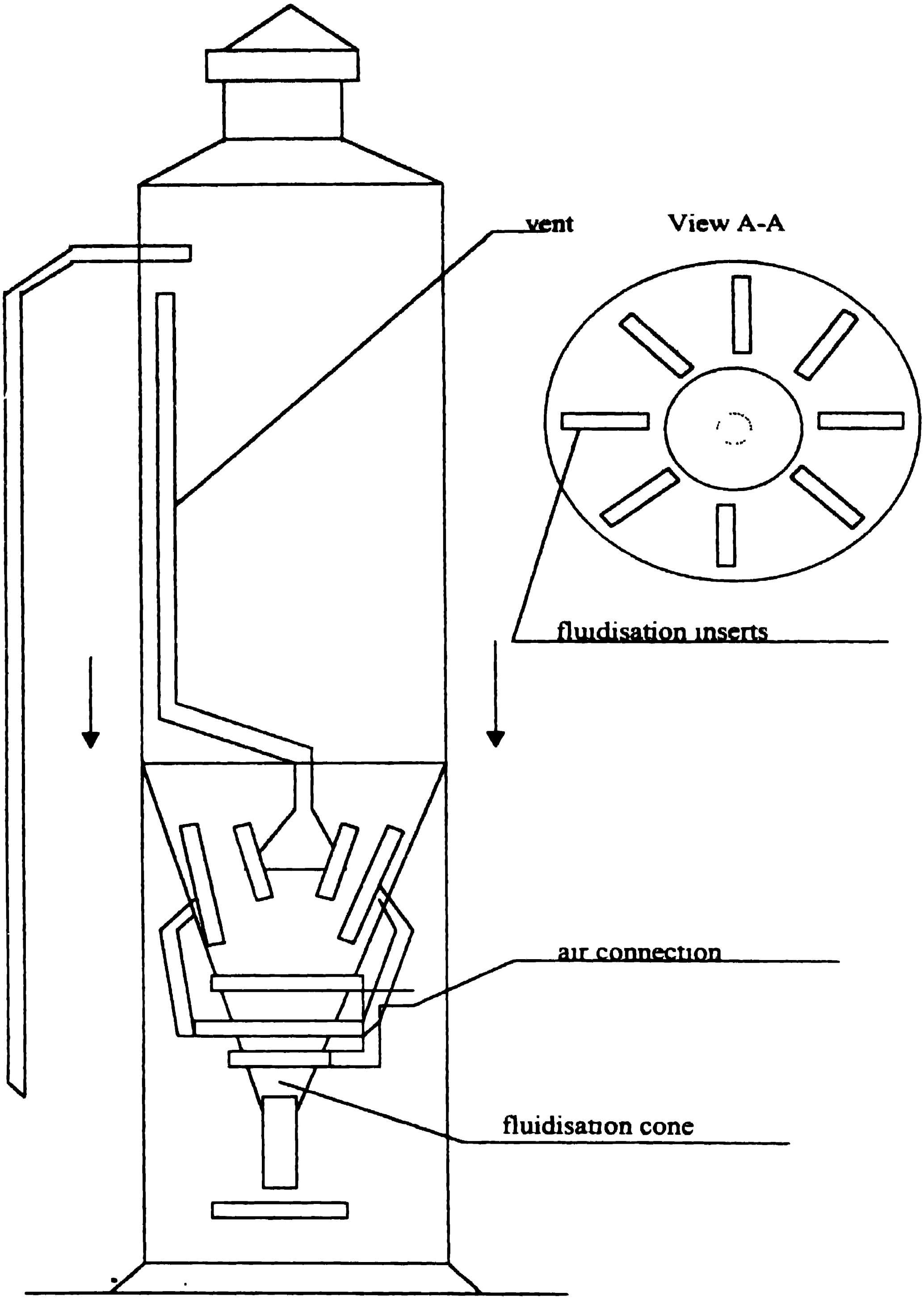
Maximum and minimum level monitors are necessary in storage silo to prevent both overfilling and unplanned emptying.

(d) Raw Material Discharge

Raw material discharge is a very important factor in the operation of a silo facility. Most conical silo outlets are built with a hopper angle of 60^0 . Except for plastics pellets, additional discharge acid must be attached to the outlet zone for virtually all fine-grained raw material. The familiar ability of many pulverulent products to flow freely when fluidised with air is exploited with the aid of aerating devices. The suitability of a product for aeration is determined by its bulk density, angle of repose, grain size distribution and specific surface area. Aeration plates are built based on a number of different systems. The surface in contact with product are made an air-permeable material. Nylon and polyester are generally used to cover the aeration plates, but air-permeable ceramic material, sintered metal, and polyethylene board are sometimes used instead.

Another important mechanical discharge aid the vibration plate. It is particularly suitable for products that tend to ' shoot over ' . In such cases, it is necessary to hold the products back while metering them to the equipment that follows.

From the storage silo , the raw material components are conveyed pneumatically to the weighing station.



Storage silo for PVC polymer or fillers

(e) Dust Removal System

The dust content of the air differs, depending on silo size, filling level and particle size of the product. Suitable filters have to be provided to remove the dust from the air. Most silo installations are laid out with a filter for each tank unit.

The filters are provided with fully automatic or semi-automatic purging, depending on the dust loading and throughput. The difference between the tow is that fully automatic filters are purged by dust-laden air during service, while semi-automatic filters are cleaned only when the filling operation is complete. Either bag or sheet filter can be used. The advantage of the sheet filter over the bag filter is that it occupies less space. For lower dust loading and coarse-particle dust, filters with mechanically actuated purging devices are generally sufficient. For very highly loaded filters and those handling find dust, jet-type filters with pneumatic purging are generally used.

Conveying of PVC Polymer and Filters

The most suitable conveying system has to be found for each material, depending on flow rate, conveying distance, and special cleanliness requirements. It is also necessary to consider the material temperature (and whether it cools down or heats up), as well as the possibility of its segregation into various fractions.

(a) Pneumatic Conveying

Pneumatic conveying, i.e. the transport of bulk materials in closed pipes with the aid of a stream of air, is standard practice in PVC processing plants just as it is in other industries. The technique has decisive advantages viz. easy adaptation of system to a given building, complete emptying of the conveying pipes, and the entirely enclosed nature of the conveying system.

CONVEYING SYSTEMS

The basic division in pneumatic conveying is between suction and pressure system. There are also combinations, which are generally known as pull / push system.

CONVEYING MODES

Flow conditions can differ widely in a conveying pipe depending on the bulk material conveying rate, air velocity, particle size, and pipe diameter. Four basic conveying modes are recognised: suspension, stream, plug, and high-density conveying.

Main features of conveying systems

System	Operating pressure range (mm w.g.)*	Compressor	Conveying distance (m)	Application	Material pick-up	Material delivery
High pressure (generally employed for high conveying rates)	15000-25000	Screw compressor	long distances	Filling of storage silos from pressure-proof tankers	Pressure-resistant silo	Cyclone with rotary valve, silo filter
Medium pressure	4000-8000	Piston compressor	200	Filling of production or holding silos from storage silos or from bag-dumping station	Rotaey valve	Cyclone with rotary valve, filter
Low pressure	<2000	Radial blower	up to 50 (very limited)	Filling of holding	Rotary valve pendulum flap	Cyclone with rotary valve filter
Suction conveyor	Vacuum of about-5000	Piston compressor	150	silo filling	Picked up by suction	(1) cyclone with rotary valve filter
	-1500	Radial blower	30	Charging pellets into bins and machines; removing free-flowing material from tips or containers		(2) Vacuum pot with filter
Suction\ pressure conveyor		Piston compressor radial blower	Short to medium distances	Silo filling, suction pick-up from grinding mills	Picked up by suction	Filter, cyclole with rotary valve

Suspension and stream conveying:

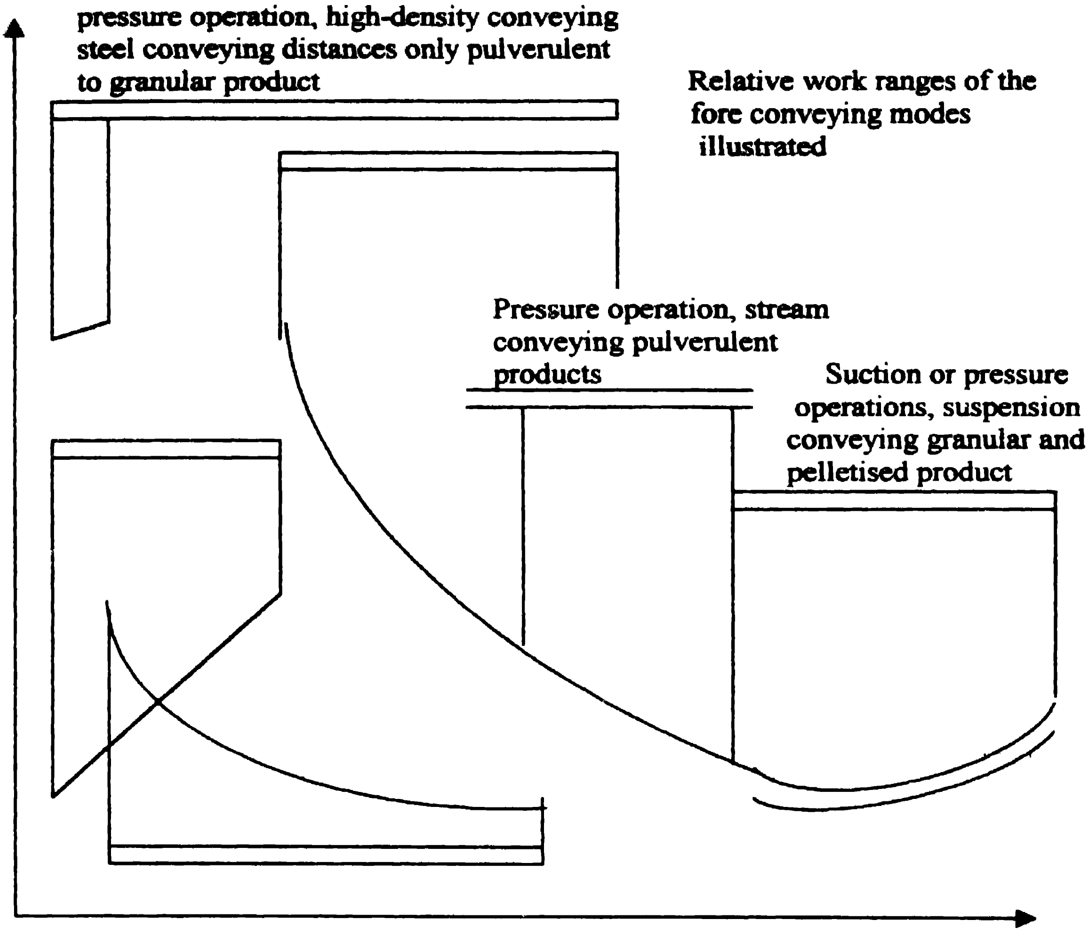
--- relatively inexpensive system,
--- virtually all pneumatically conveyable products can be handled,
--- conveying usually remains effective when products are modified,
--- appropriate in cases where on extreme demands arise in terms of operating time or conveying rates.

Low-velocity conveying:

--- gentle treatment of material conveyed,
--- avoidance of abrasion (dust, floss),
--- low system wear,
--- low energy consumption,
--- low air consumption,
--- low dust removal cost,

		Conveying state	Velocity		Solids loading	pressure drop	
			air w $\left(\frac{m}{s}\right)$	material Wp $\left(\frac{m}{s}\right)$	u^{a} (-)	p $\left(\frac{bar}{m}\right)$	
	1	suspension conveying coarse material	W>Ws 20 - 35	(0 5 - 0 8) W	< 30	0 1-1	
		Suspension conveying fine material	W>Ws 15-30	(0 9-1) W	<10	0 1-1	
	2	Stream conveying fine material	W>Ws 10-25	particles 09 - 1W streams (0 1-0 3)W	10-30	1-3	
		Streams & lumps, unstable range commercommercially unstable	5-15	particles (0 9-1) W lumes ca0 7 W	10-30	un defined	
Low-velocity conveying	3	Fluidised conveying lose line material	W>˂Ws 3-10	-W	-150	-1	Air-impermeable products
		Plug conveying	W>Ws 3-15	(0 5-0 8) W	20-150	0 5-6	
	4	High- density conveying	W>` Ws 0 5-8	-W	-150	-6	Air-permeable products
		Pulse conveying (intermittent high density conveying)	W >´ Ws 2-10	-0 6 W	-150	0 5-6	

Typical conveying modes in pneumatic conveying systems All figures refer to a pipe of 100 mm nominal size


pressure operation, high-density conveying steel conveying distances only pulverulent to granular product
Relative work ranges of the fore conveying modes illustrated
Pressure operation, stream conveying pulverulent products
Suction or pressure operations, suspension conveying granular and pelletised product

Variable dependent on conveying gas $Fr = \frac{w^2}{Dg}$

curve of equivalent conveying rate . Generalised representation Fr = Froude's number (a dimensionless parameter) ; W= air velocity (m s^{-1}), D= pipe diameter (m); g= gravity constant (m s^{-2})

--- stable conveying mode,
--- low noise level along pipes and at separator,
--- no serious problem with electrostatic charges.

The conveying modes usually employed in PVC processing plants are the following :

E-PVC: Mostly suspension conveying, but some plug conveying, because the fine powder can not stick to the pipe walls when so conveyed. In the case of suspension conveying, the solids loading is

$$10 \frac{\text{kgh}^{-1}\ \text{solid}}{\text{kgh}^{-1}\ \text{air}}$$

S-PVC: Mainly suspension conveying; solids loading is

$$20 \text{ to } 25 \frac{\text{kg h}^{-1}\ \text{solids}}{\text{kg h}^{-1}\ \text{air}}$$

Filler: Mainly suspension conveying, though for abrasive materials such as untreated chalk, plug conveying is possible with loadings of

$$60 \text{ to } 80 - \frac{\text{kg h}^{-1}\ \text{solids}}{\text{kg h}^{-1}\ \text{air}}$$

PVC pellets: Suspension conveying, though fluidised conveying (low velocity) is more effective in avoiding pellet abrasion. The nominal size of 80 and 125mm are normally made of stainless steel.

Storage of plasticisers

(a) Tank sizes

The installation of storage tanks can become an economical proposition at consumption levels of about10 tonnes per month of a plasticiser for the following reasons:

---- lower purchase price for plasticiser,
---- easier product handling,
--- labour savings,
--- less storage space required,
--- easier survillance of stock.

(b) Suitable Construction Material

Tanks

---- corrosion- risistant steel;

---- steel with paint finish resistant to the plasticiser;
---- steel, sand-blasted, internal walls sprayed with plasticiser;
---- aluminium;
---- glass-fiber reinforced Palatal.

Pipes

---- corrosion-resistant steel;
---- steel;
---- aluminium;
---- glass-fibre reinforced Palatal

Valves, ball valves

---- corrosion-resistant steel;
---- steel;
---- aluminium;
---- cast iron.

The pumps should be equipped with single-acting mechanical seals, because packing cords are attacked by the plasticiser

(c) Plasticiser Delivery

Small consumers normally receive the plasticiser in drums, and large-scale production plant in road tankers holding 10-20 tonnes or in rail tank car holding between 15 and 30 tonnes.

Road tankers normally have an outlet nozzle of 80 mm nominal size, and are equipped with a pump or a compressor for emptying the plasticiser. Maximum delivery head is 10 m, provided the piping is straight.

(d) Pointers on pipe laying

All pipe should be laid so that it will run empty into the storage tank. It is advisable to use 80 mm nominal size piping for empyting the storage tanks.

Storage of Additives

processing additives are delivered, and subsequently stored in the processing plant, in small containers such as drums, bags or like. Additives required in the product line are emptied at dust free bag or drum-dumping stations into the production. The additives are generally withdraw with the aid of metering screws, which feed them into the material flow as required by particular formulations.

Metering and Weighing

(a) Fundamentals of Metering and Weighing Technology

If a meterial must be removed from a silo or tank at a steady rate, or if a specified weight or volume must be removed, metering equipment is required. Accurate metering of the individual raw meterials is a crucial prerequisite of precise formulation maintenance, and therefor of final product quality. Generally, the raw materials and the final product are the factors determining whether to meter gravimetrically or volumetrically, continuously or intermittently.

CONTINUOUS VOLUMETERIC METERING

The following volumetric metering equipment is available for raw material components in powder or paste form, depending on the physical properties of the product.

--- Metering screws (in single- or twin-screw designs): Single screws are employed in the form of simple helical screws, helical ribbons and worms, sometime with supplementary vibration or concentric spirals in the inlet to break up the product:
--- Belt metering devices:
--- Vibrating chutes:
--- Rotary valves:

Liquid raw material components are metered by means of positive displacement pumps (piston or gear).

CONTINUOUS GRAVIMETRIC METERING

In the case of gravimetric metering - and in contrast with volumetric metering- neither the various product properties, such as particle size, particle shape, bulk density or flow behaviour, nor the specified throughput rate, make any difference whatever.

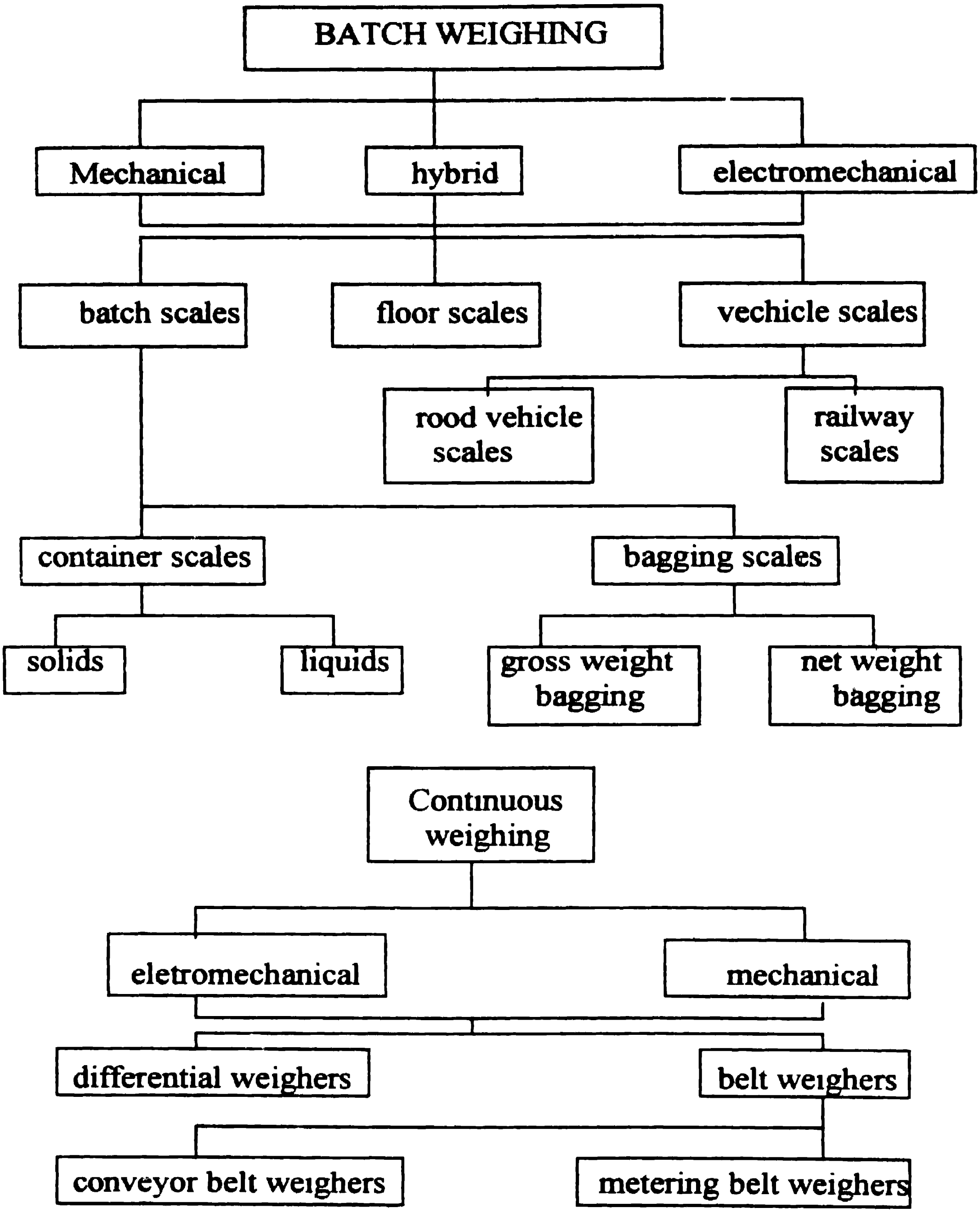
The operating principle of a differential metering weigher is totally different from that of the continuous belt weigher or metering screw weigher.

BATCH WEIGHING SYSTEMS

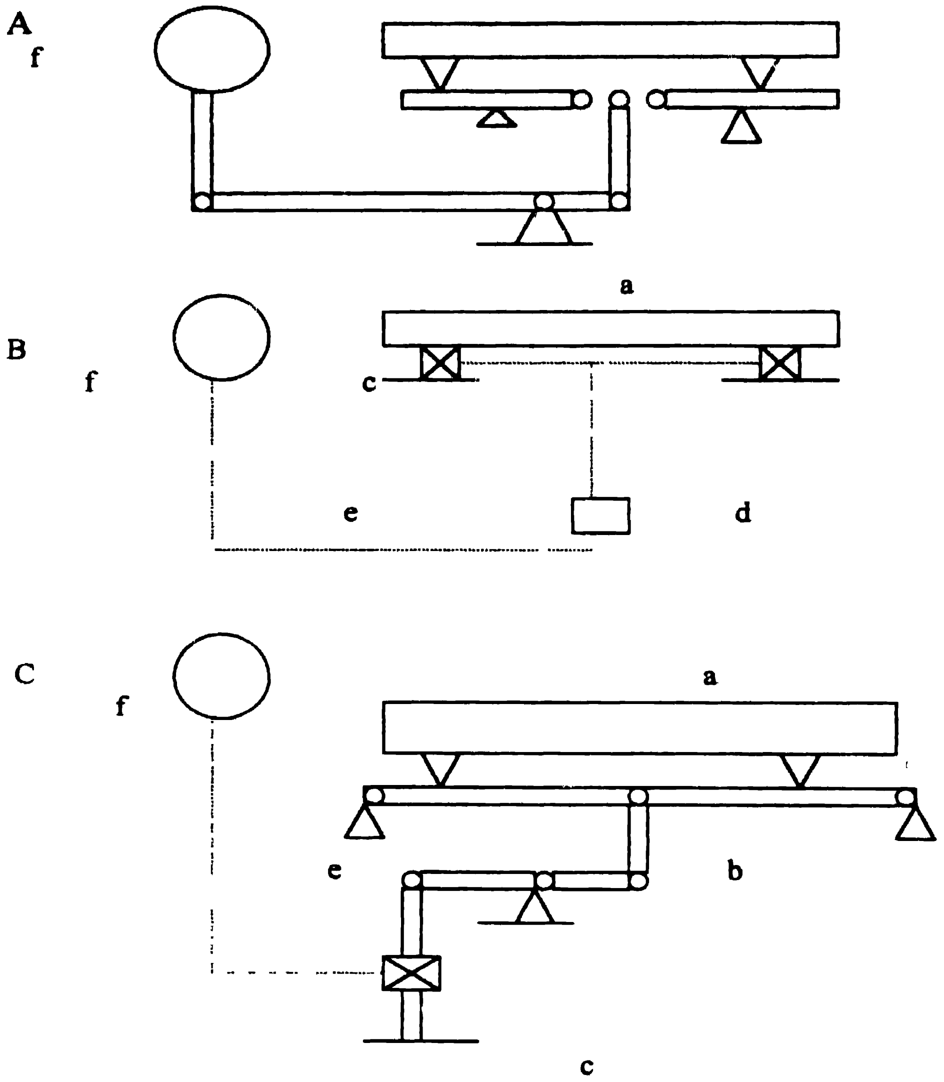
--- digital display of weight readings.
--- handling separated from the weighing station.
--- automatic control functions and calculations with the use of microcomputers,
--- data processing and display unit that can be placed anywhere.
--- compatibility with peripheral computer and data processing installations.

Solid raw material components: in this operation, the smallest possible batch metering error is determined primarily by the accuracy of the container scale. The only demands placed on the scale charging equipment are that it should deliver a roughly continuous flow of bulk material, and that this should be cut of virtually instantaneously when the preset weight is reached. The metering accuracy of the piece of equipment used to charge the scale has no influence on the batch metering error. Container scales are charged with volumetric metering devices such as rotary

valves, vibrating chutes, single and double metering screws, metering screws with agitators, or vibrating screws



Batch and continuous weighing systems



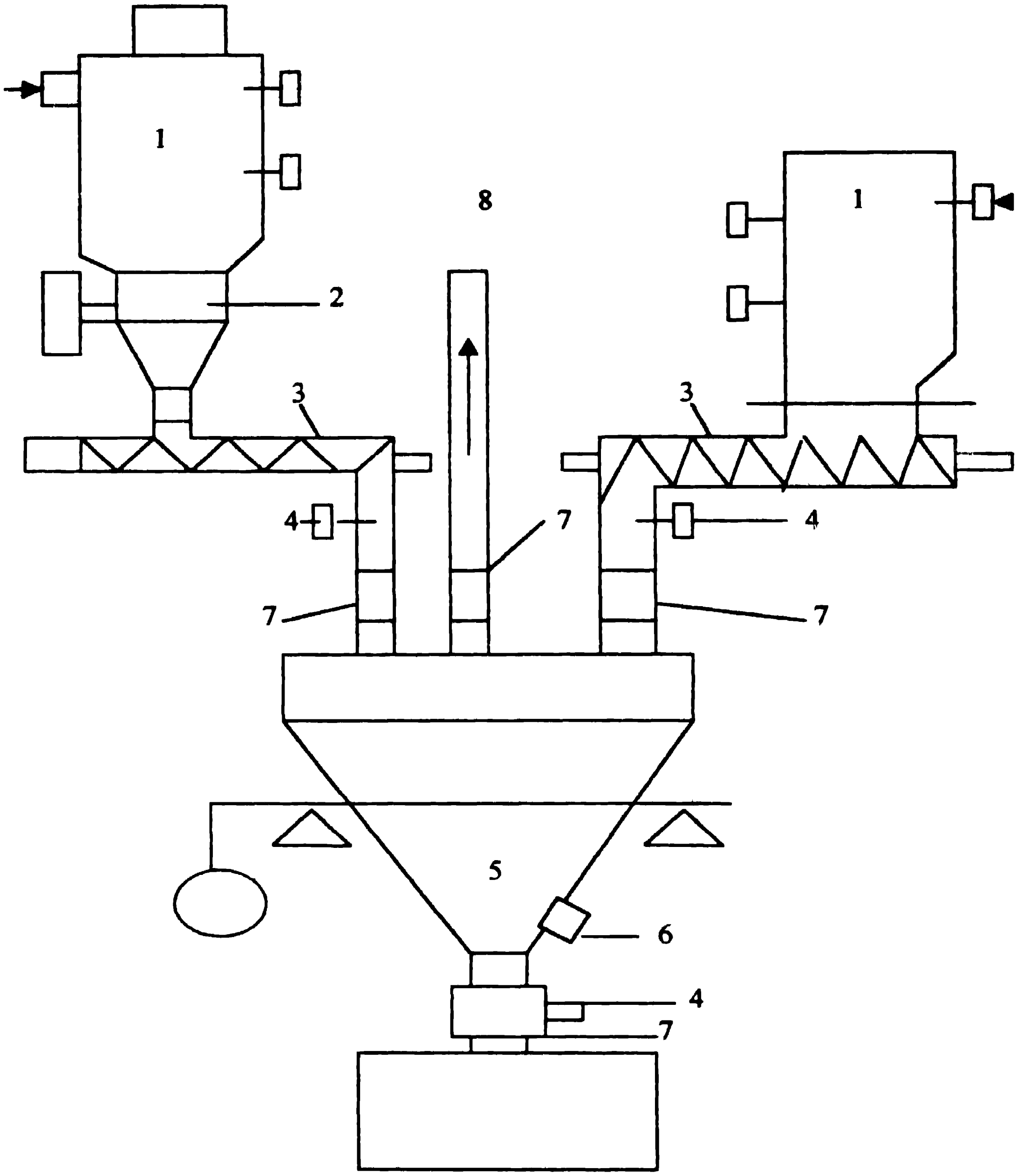
Weighing systems. A. mechanial: B, electoomechancal ; C, hybrid, Key: a, load reacptacle; b, lever system ; c, force detector; d, junction box; e, cable; f, weight display.

Many weighing errors are traceable to incorrectly installed scale containers. Every scale container must be able to move freely in the vertical direction.

The entire process-- metering of the raw meterial components in a specific order, weighing, emptying of the scale into a solding container or preparation for pneumatic conveying or emptying of the scale directly into the primixer, initiation of the mixed weighing process, batch counting, and finally the puenumatic refilling of the production or holding bins from the storage silo-runs completely automatically.

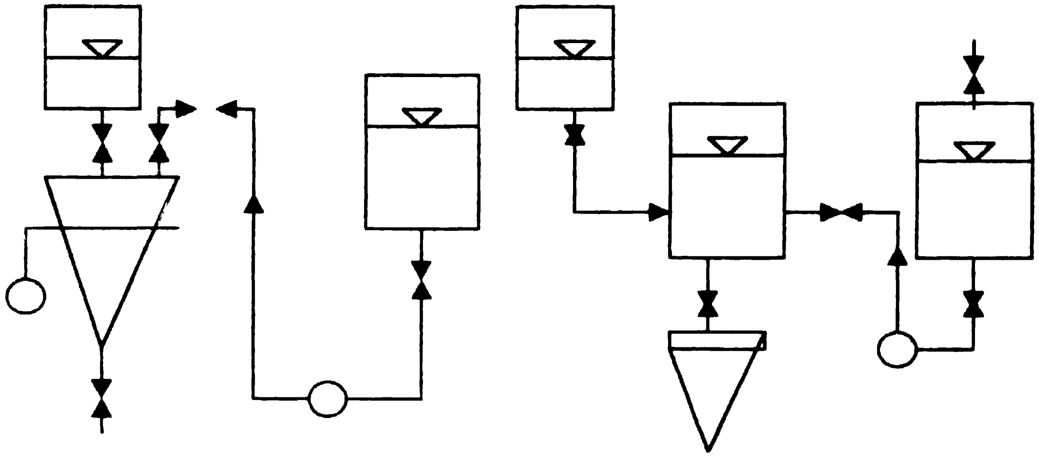
The batch weighing installation is rightly called the 'heart of the overall production line.

Liquids: In this case the material being metered flows by gravity out of a storage tank into the scale tank , or else is pumped into it from a lower level. Temperature, density and viscosity fluctuation have no effect on metering accuracy here. Highly viscous liquid are usually heated.



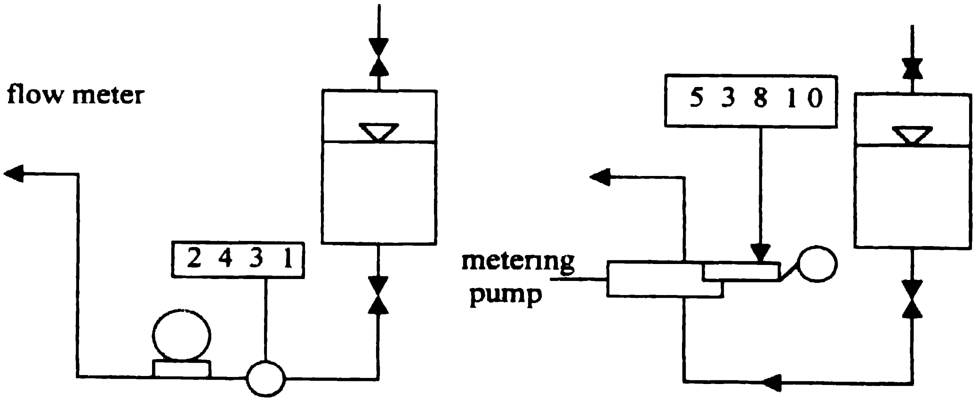
Weighing system for solids 1. Container for solids; 2. discharge plate; 3. metering screw, 4. pneumatic closure flap, 5. container scale; 6. beater. 7. elastic collar; 8. vent.

Where liquids are metered by volume, they are again conveyed by gravity or a pump into a collecting tank. In this case, the feed is cut off when a certain filling level is reached. This is a very simple metering method, but density fluctuation result in metering errors. Flow measurement, is based on measuring the volume of liquid delivered by a pump. Because most flow meter react sensitively to fluctuations of the liquid's viscosity, metering installation of this type are sometimes heated up to a constant temperature.



A Weighing

B. volumetric measurement

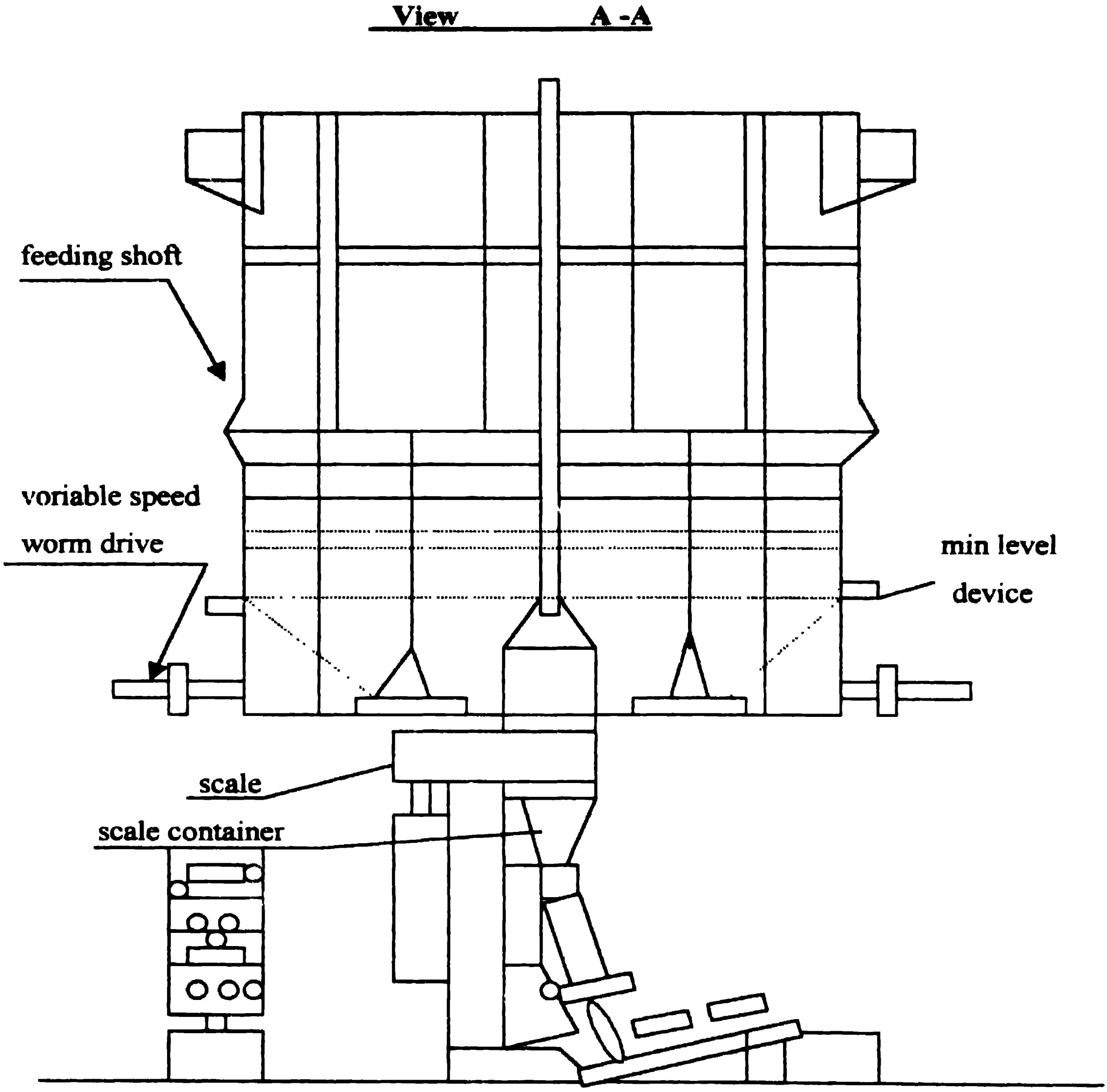


C Flow rate measurement

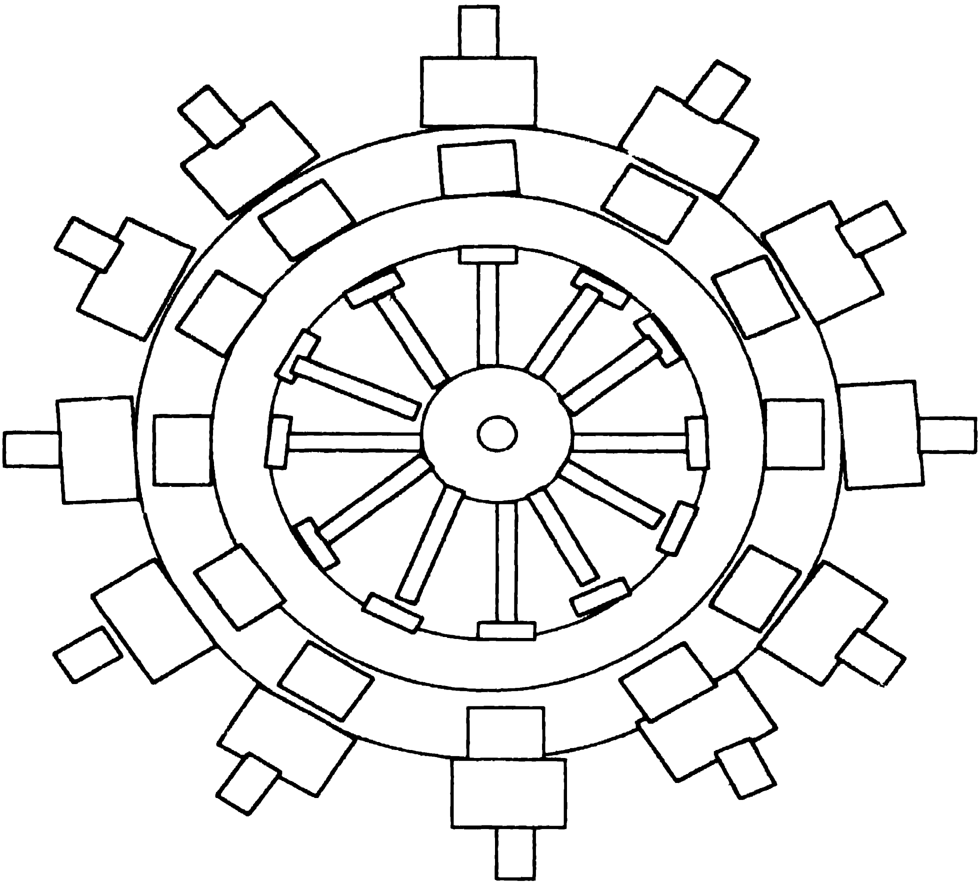
D Volumetric displacement

Metering methods for liquids

Minor additives : These are the additives whose quantities assembled for each batch are smaller than the quantity of PVC polymer by one or two powers of ten i.e. their total content in the overall formulation is not more than 10 % (and commonly 0.5 - 5%). For this reason, the additives are handled in a number of different ways in the PVC compounding field. Often they are weighed by hand by one person and then filled individually into PVC pouches for incorporation into the particular composition.



fully automatic weighing installation for additives



fully automatic weighing installation for addttives

(b) Control and Monitoring Equipment

SYSTEM ACCURACY IN COMPOUNDING PLANTS

Even in a thoroughly plenned metering installation, sizeable deviations can occur under certain conditisons.

Very roughly state, the following sources of error exit in the individual functions functions of the control loop:

— mechanical errors in the scale,

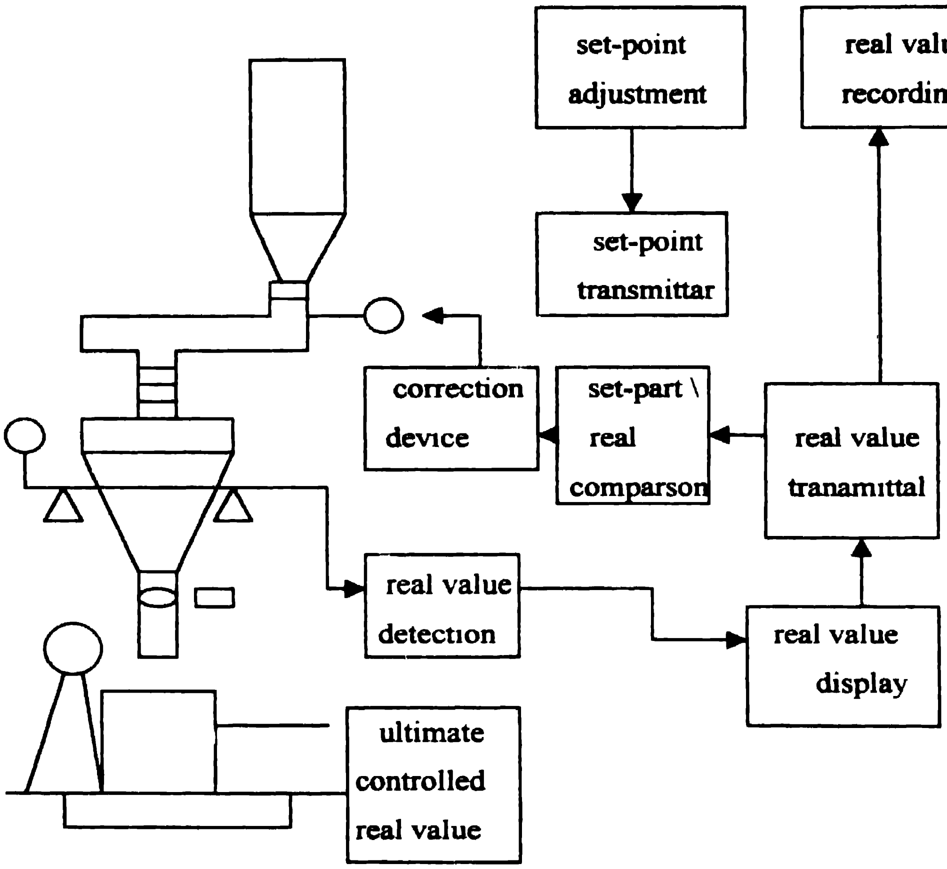
--- cut-off errors in metering,

--- discharge errors during emptying of the scale,

--- errors in data input or acquistion.

BATCH ASSEMBLE

As illustrated the control requirements for the actual weighing of the components are relatively modest. The output singnals coming from the weighing installation comprise only the momentary weight readings in digital or analogue from. The only input signals required are the variables acting on the positioning elements, MI and M2, and on the closing device, M3, of the scale container. Only tow connections are required for the set- point input and the real value recording.



Control loop of a weighing installation

The metering procedure in a plant designed along modern lines can run as follows:

--- Switch on plant in general.

--- Set all scales to 'automatic' If necessary, zrro taring can be carried out from the control desk. Automatic starting is only possible when all scales are at 'zero'.

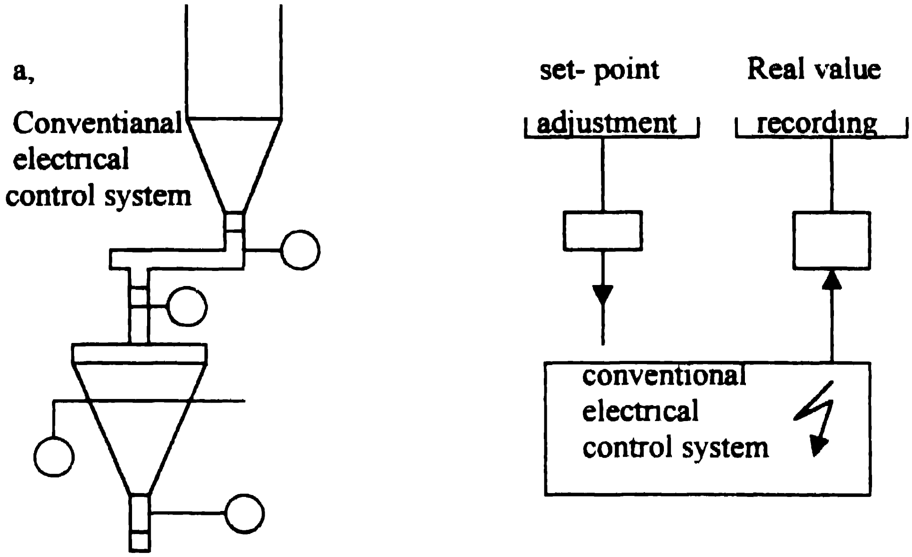
--- Place punched cards in the reader.

--- Release mixer filling system.

--- Formulation is called for by the mixer control system

--- Metering of the individual components in coarse \ fine stream with 'finished' report following the last components.

--- Emptying of the individual scales in predetermined order and with predetermined timing.


a,
Conventianal electrical control system
set- point
adjustment
Real value
recording
conventional electrical control system

Control equipment for container scales

---- Transfer of batches (solid components, liquids, additives) to the mixer calling for them.

---- All scales move to zero and the next weighing procedure can be inittated.

analog and digital remote displays at the control desk make it possible to supervise the weighing installation.

Compounding: Operations and Equipment

General

The function of compounding is to combine the PVC polymer with the other components of the formulation and to provide the resulting composition (compound) in a form appropriate to the further processing intended. In laboratory compounding the form may have to be suitable for testing or experimentation.

Industrial PVC compounding operations may be grouped under the following general headings

(a) Preparation of powder compositions (pre-mixes and dry blends).
(b) Melt-compounding ,with production of pelletised compositions.
(c) Compounding for direct feeding calenders.
(d) Preparation of liquid compositions -pastes, latex compositions, and solutions.
(e) Recycling (of own-process scrap of laimed material).

The equipment used in industrial compounding of PVC can be divided in to the upstream section a head of the compounding, the compounding section, and the downstream section. The upstream equipment is much the same in all the five types of compounding operation, but the compounding set-up and corresponding down stream equipment vary according to operation type.

By virtue of their small scale, laboratory compounding operations do not normally require any elaborate or automatic upstream equipment. The compounding equipment is either a reduced-size version of the corresponding industrial machinery (with two-roll mins in much wider use in the laboratory then they are in industry now a days), or special instrumented apparatus based on the torque rheometer (as represented for example by the widely used bender equipment is also common simples then its industrial counter parts.

INDUSTRIAL PRODUCTION OF SOLID COMPOUNDS

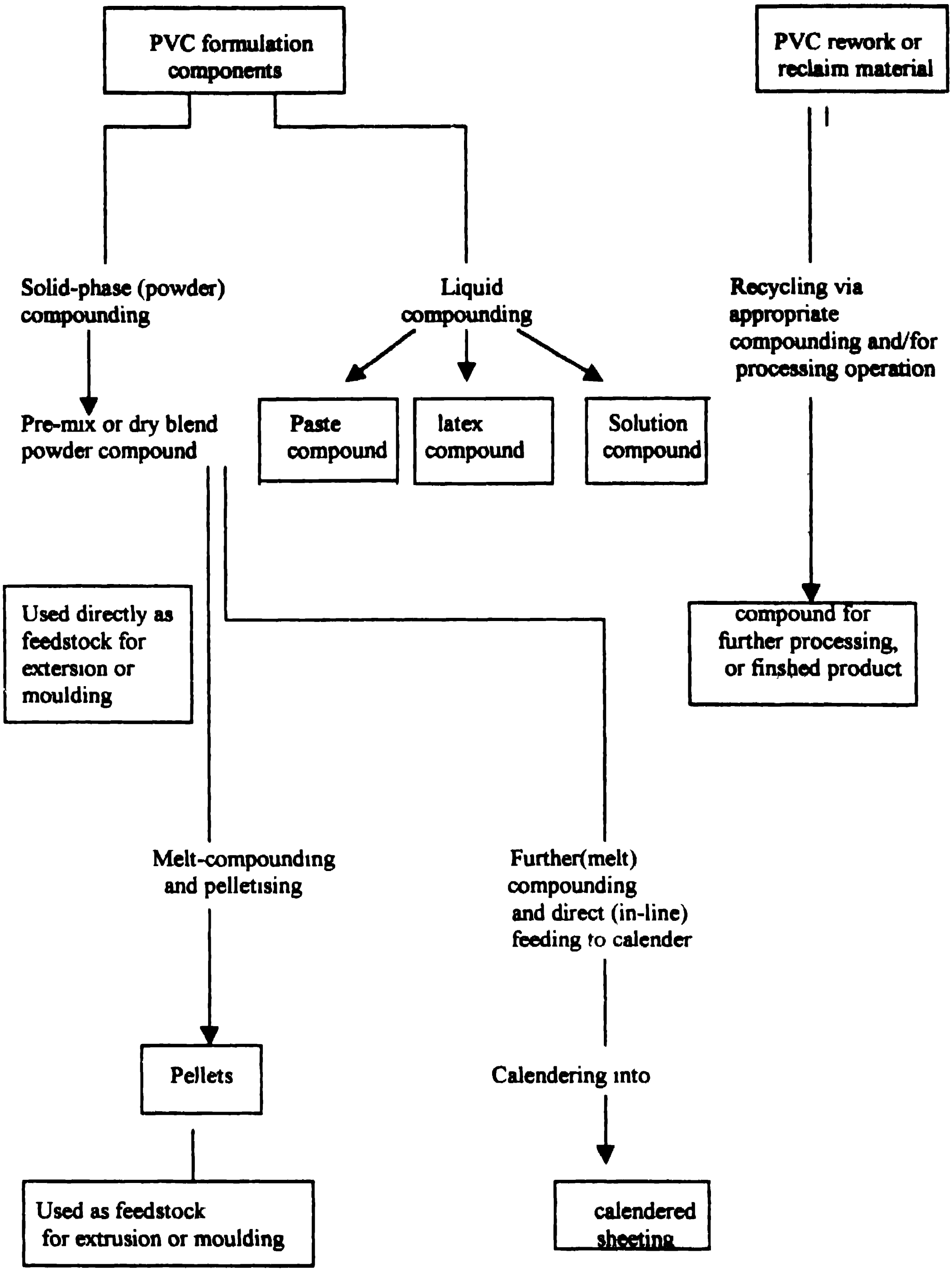
Upstream Equipment and operations

(a) Handling, Storage and Conveying

POLYMER AND FILLERS

In relatively small-scale industrial operations, these powder materials may be delivered in containers or bags, which are then kept in suitable stores . Otherwise, delivery is by tanker (road or rail) and silos are used for storage (typically where consumption is about 30 tonnes or above, per month, per material. Silo capacities fairly typically range between 150 and 250 m^3, although units outside this range are also employed in some cases. Common construction materials are an aluminium /

magnesium alloy, or glass fiber-reinforced polyester (for silos near the lower and of the above capacity range).


PVC formulation components
PVC rework or reclaim material
Solid-phase (powder) compounding
Liquid compounding
Recycling via appropriate compounding and/for processing operation
Pre-mix or dry blend powder compound
Paste compound
latex compound
Solution compound
Used directly as feedstock for extersion or moulding
compound for further processing, or finshed product
Melt-compounding and pelletising
Further(melt) compounding and direct (in-line) feeding to calender
Pellets
Calendering into
Used as feedstock for extrusion or moulding
calendered sheeting

Compounding of PVC : General schematic outline

Pneumatic conveying systems (normally pressure rather then suction-operated) the use of fill silos from road or rail tankers; such vehicles are designed to withstand the pressure involved. Screw compressors are used to generate the required flow of oil free air. With the usual pipe diameters of 80-100 mm, typical conveying capacities are in the range 15-30 tonnes per/hour. Control systems are available for silo and tank filling levels, providing visual displays and printout (e.g. equipment from Keller Spezialtechnik, FRG).

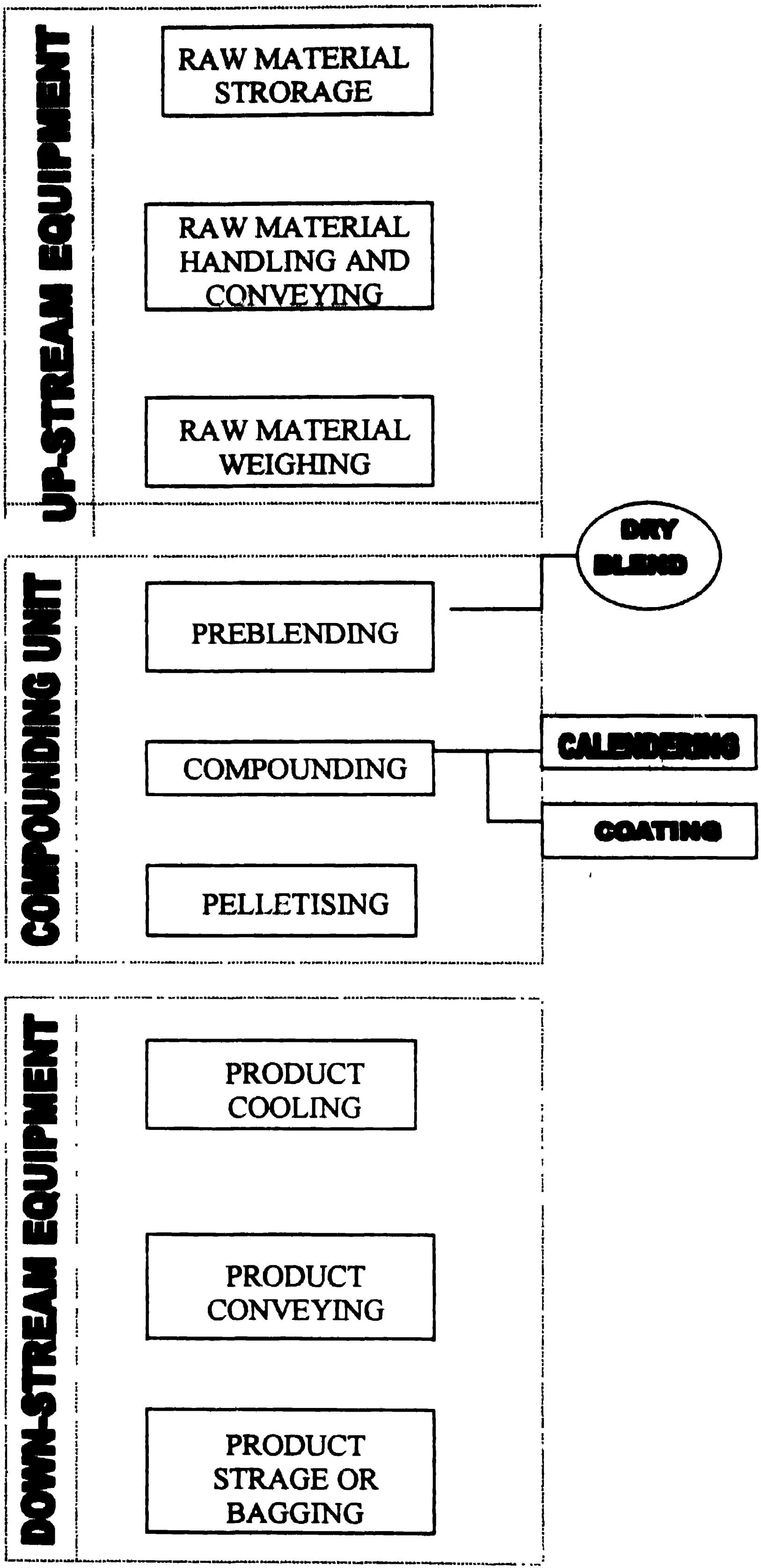
For operational flexibility and ability to handle smoothly small batches of different formulation and qualities if required, most plants have an additional dumping station for filling the storage silo(or holding bins, or booth) with bag -packaged goods. A manual dumping station (hand opening and dumping of bags), with a filling rate of -typically-about three tonnes per/hour, is often sufficient. Where larger quantities of bagged material have to be handled, a semiautomatic or automatic bag-dumping machine may be needed (typical capacity about 6000 bag per/hour).

Bag dumping stations must include provision for drawing off dust airborne during dumping to a filter via a suitable exhaust system. As rule , this station are supplied with a built-in inter which can be cleaned mechanically to return the collected dust to the bulk material. An exhaust system should provide an air flow of about $20m^3$ min^{-1} This resulting are withdrawal velocity during dumping should be about0 5m s^{-}

Discharge in an important operation in the functioning of a silo. Most conical silo out lets are built with a hopper angle of 60^0. Except for pellets, virtually all fine particulate material required discharge aids. Fluidisation of polymer and filler powders is widely use to add flow· the powder is fluidised by blowing air (which must be free of dust and oil) through aeration plates (covered with nylon or polyester fabric, porous ceramic or sintered metal plates, and some times polyethylene board). The plates are designed to produced a pulsation effect by blowing in the air section wise. Another important mechanical discharge aids is the vibration plates, particularly useful in restraining materials that tend to `shoot over'.

Pneumatic filling of silo result in dust-loading of air, differing in extent depending on silo size, filling level, and powder particals size The dust is removed by suitable filter units (separate ones for each tanks, except where a number of silos are permanently used to store the same material).

Any two consecutive operations in the pneumatic sequence for solid compounds are usually separated by a transport distance to be traversed blends by the material concerned (solid starting components powder blends, finished pellets, rework or reclaimed material for recycling). The available conveying system are of two type : pneumatic and mechanical (employing screws, bucket elevators, etc.), and the type and its most suitable variant is selected in the light of such considerations as the conveying distance, transport rate required special cleanliness requirements, material temperature (and whether it will cool or heat up in transit), and possibility of segregation in to fraction.



PVC compounding : block diagram

Pneumatic conveying, i.e. the transport of bulk material in closed pipe with the aid of a stream of air, is standard practice in PVC processing plants just as it is in other industries. The technique has decisive advantages, easy adaptation of the system to a given building, complete emptying of the conveying pipes. and the entirely enclosed nature of the conveying system. The basic division in pneumatic conveying is between suction and pressure systems. There are also combinations, which are sometime referred to as pull / push system.

Flow conditions can differ widely in a conveying pipe, depending on the bulk material conveying rate, are velocity, particle size, and pipe diameter. Four basic conveying modes are recognised : suspension, stream, plug, and high-density conveying.

Traditional systems work on the suspension principal, with air velocities between 15 and 30 m s^{-1} and material loading upto 30 kg of material per kg of air throughput.

Typically, the individual particles are distributed virtually evenly over the pipe cross-section and ricochet from one side of the pipe to the other while being conveyed.

At air velocities in the range 5-15 m s^{-1}, the material no longer ricochet; instead, they are pushed through the pipe in the from of lumps and plugs. Some of their respective basic features may be listed follows.

Suspension and stream conveying : Relatively inexpensive system; suitable for virtually all pneumatically conveying material tolerant of material modifications: appropriate where no extreme demands arise in terms of operating time or conveying rates.

Low- Velocity conveying · Gentle treatment of material conveyed stable conveying ; no excessive abrasion (generating dust or floss); no serious problem with electrostatic charges system wear energy consumption, dust removal cost, and noise level along and at separator, all low.

The conveying modes usually employed for solids, particulate material in PVC processing plants are summarised.

PLASTICISERS

Small-scale processors normally receive plasticisers drums (per 200 kg), and large- scale ones in bulk, in road-tankers holding 10- 20 tonnes (occasionally in tanker compartments holding 5 tonnes) or in rail-tank cars of 15-30 tonnes capacity.

It is usual for road-tankers to have an outlet nozzle of 80 mm nominal size and to be equipped with a pump or compressor discharging the plasticisers. The normal maximum delivery head is 10 m., providing the piping is straight. Most rail-tank cars have outlet nozzles of 100 mm nominal size. Here the customer must provide

the pump for emptying the car. A properly dimensioned pump will drain a tank car in about one hour. The storage tank should be installed in the immediate vicinity of the siding. All delivery pipe work should be laid so that it can run empty into the storage tank. It is advisable to holding or production tanks. Heating (especially for pipelines) may be necessary to reduce plasticiser viscosity, and hence edge the flow (particularly with highly viscous plasticisers, and / or in cold weather).

Main features of conveying systems

System	Operating pressure range (mm w g)*	Compressor	Conveying distance (m)	Application	Material pick-up	Material delivery
High pressure (generally employed for high conveying rates)	15000-25000	Screw compressor	long distances	Filling of storage silos from pressure-proof tankers	Pressure-resistant silo	Cyclone with rotary valve, silo filter
Medium pressure	4000-8000	Piston compressor	200	Filling of production or holding silos from storage silos or from bag-dumping station	Rotaey valve	Cyclone with rotary valve, filter
Low pressure	<2000	Radial blower	up to 50 (very limited)	Filling of holding	Rotary valve pendulum flap	Cyclone with rotary valve filter
Suction conveyor	Vacuum of about-5000	Piston compressor	150	silo filling	Picked up by suction	(1) cyclone with rotary valve filter
	-1500	Radial blower	30	Charging pellets into bins and machines, removing free-flowing material from tips or containers		(2) Vacuum pot with filter
Suction\ pressure conveyor		Piston compressor radial blower	Short to medium distances	Silo filling, suction pick-up from grinding mills	Picked up by suction	Filter, cyclole with rotary valve

		Conveying state	Velocity		Solids loading	pressure drop	
			air w $\left(\frac{m}{s}\right)$	material Wp $\left(\frac{m}{s}\right)$	u^{a} (-)	p $\left(\frac{bar}{m}\right)$	
	1	suspension conveying coarse material	W>Ws 20 - 35	(0.5 - 0.8) W	< 30	0.1-1	
		Suspension conveying fine material	W>Ws 15-30	(0.9-1) W	<10	0.1-1	
	2	Stream conveying fine material	W>Ws 10-25	particles .09 - 1W streams (0.1-0.3)W	10-30	1-3	
		Streams & lumps, unstable range commercommercially unstable	5-15	particles (0.9-1) W lumes ca0.7 W	10-30	un defined	
Low-velocity conveying	3	Fluidised conveying lose line material	$W >^{<} Ws$ 3-10	-W	-150	-1	Air-impermeable products
		Plug conveying	W>Ws 3-15	(0.5-0.8) W	20-150	0.5-6	
	4	High- density conveying	$W >^{<} Ws$ 0.5-8	-W	-150	-6	Air-permeable products
		Pulse conveying (intermittent high density conveying)	$W >^{<} Ws$ 2-10	-0.6 W	-150	0.5-6	

Typical conveying modes in pneumatic conveying systems. All figures refer to a pipe of 100 mm nominal size .

Pneumatic Conveying of Solid, Particulate Materials in PVC Processing: Conveying Modes and Solid Loadings

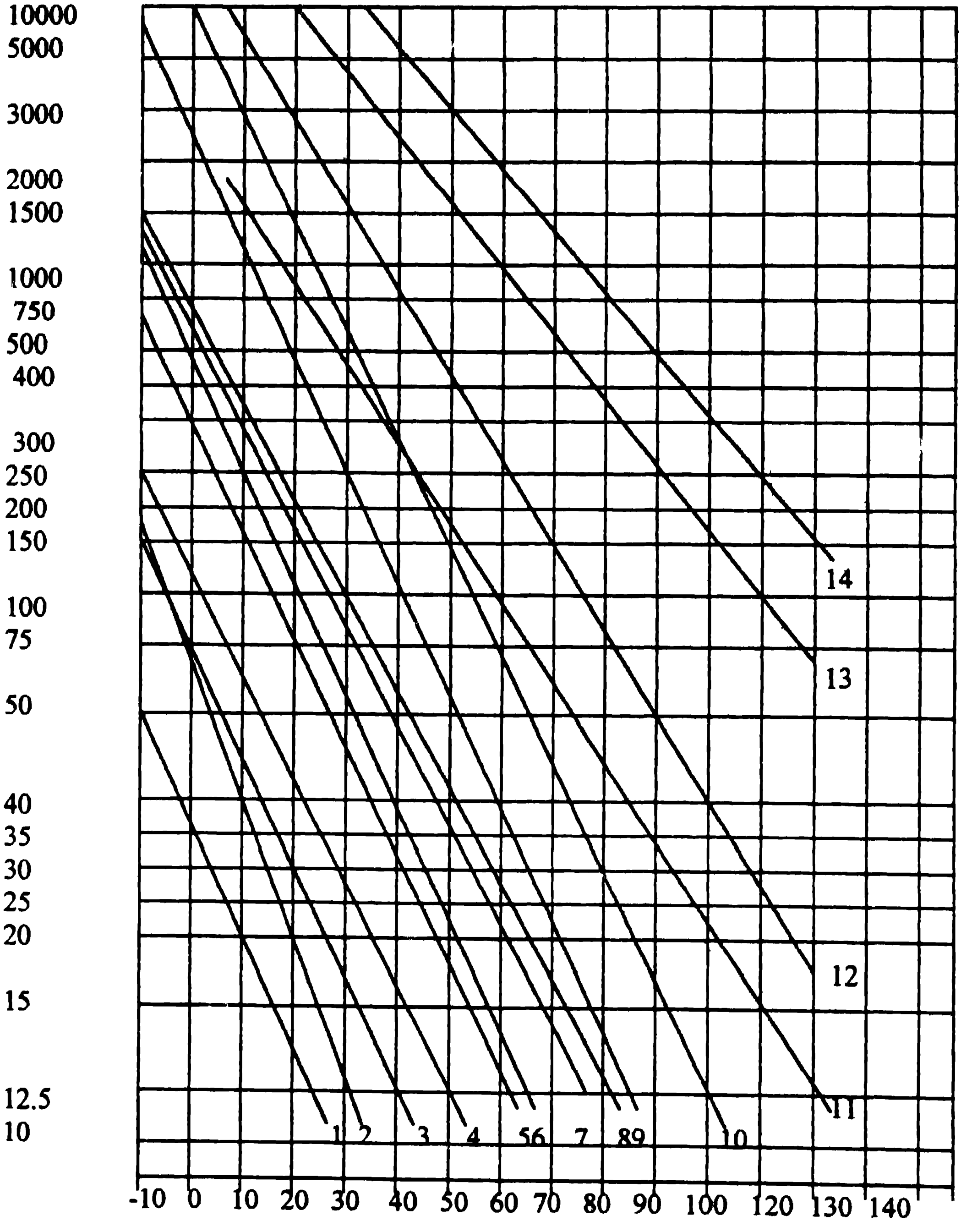
Materials	Conveying Mode	Typical solids loading (kg h -1 solids : kg h -1 air
PVC polymer (suspension-or mass -type)	Mainly suspension conveying	20-25
PVC polymer (emulsion type)	Mostly suspension, but also some plug conveying (because the fine powder cannot stick to the pipe walls in this mouds	about 10
PVC pellets	mainly suspension, although fluidised conveying (low-velocity) more effective in avoiding pellet abrasion	20-25 (suspension); about 150 (fluidised)
Fillers	Mostly suspension conveying with abrasive materials (e.g. untreated chalk) plug conveying possible, with solids loading shown	10-25 60-80

A storage tank should be large enough to accommodate the entire contents of one delivery vehicle. It is advisable it install a second tank, to ensure continuous withdrawal and complete emptying. Typical storage tank capacities are 6, 12, or 24 m ^{3}. Tanks may be constructed of corrosion-resistant steel, steel with paint finished resistant to the plasticiser, aluminium, or a suitable grade of glass-reinforced polyester resin (GRP). Suitable pipe and valve construction materials include corrosion-resistant steel, aluminium, GRP (for pipes), and cast levels (of each plasticiser) above about 5 tonnes per month.

OTHER FORMULATION COMPONETS (ADDITIVES)

These are delivered, and subsequently stored in the processing plant, in small containers such drums, bags or the like. Additives required in the production liner are emptied at dust-free or drum-dumping station into the production or holding silos with capacities between 0.5 and 2 m^3. These silos are normally made of

stainless steel. The additives are generally withdrawn with the aid of metering screws, which feed them into the material flow as required by particular formulations. The various types of conveying screws were recently reviewed by Hoppe & Eberharth.2



Viscosity-temperature relationship of a range of plasticiser

(b) Metering

GENERAL

Where, as is normally the case, material has to be removed from storage (e.g. silo, tank, etc.) in specified amounts (by weight or volume) or at a specified constant rate, metering equipment is used. Accurate metering of individual formulation components is cardinal to the maintenance of composition constant and hence of final product quality. The nature of the components and the final product are the main factor determining the metering method to be used.

The accuracy achievable is also an important consideration, especially where relatively small batches may have to produced to formulation calling for small proportions of some of the components. The general kinds of metering method available for choice are volumetric metering (continuous or intermittent) or gravimetric metering (continuous or intermittent).

The accuracy of continuous volumetric metering is effected by variation in physical properties of the materials metered (bulk density, particle size and shape, and flow behaviour of powders; density and viscosity hanges of liquids with temperature). In weight terms (which is the basis of the formulation and assembly of PVC compositions) this sensitivity limits the accuracy of volumetric metering to about + 2-5 %, depending on the materials and equipment concerned.

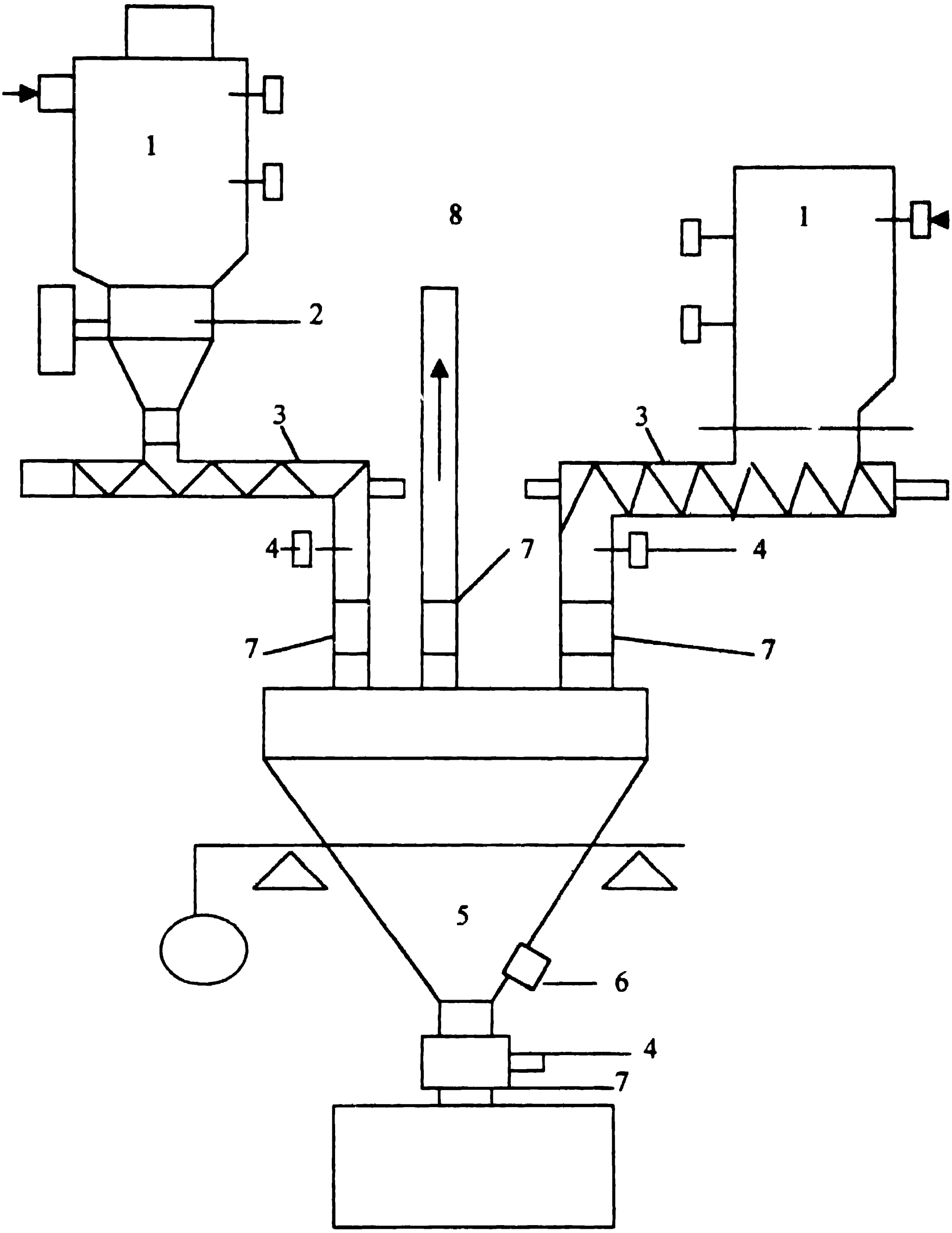
Continuous metering by weight is not sensitive to the effect of material properties, and accuracy (short-term deviation from set points) within ± 0.3-0.5 % is achievable, e.g. with belt weighers or metering - screw weighers.

MAYOR FORMULATION COMPONENTS

Batch weighing is the metering method widely used for these components in modern PVC compounding plants in which batch wise production of powder compounds (pre-mixes, dry blends) is practised. *Inter alia*, batch-weighing effectively utilises the advantages offered by the high accuracy of modern weighing equipment. The weighing systems may be mechanical, electromechanical, or hybrid types.1 The batch-weighing installation is an important section of modern PVC compound product line.

Most such lines have at least to installations of this kind. One, for weighing solid components, is integrated into the automatically operated sequence comprising tering of the formulation components in a specific order, weighing (set-point / actual comparison), emptying the scale into a holding container or directly into a pre-mixer, initiation of the mix-weighing process, batch counting, and pneumatic

refilling of the production or holding bins from the storage silos. The other main weighing installation is used for metering plasticisers, which are circulated by gear-type pumps from the production of holding tanks via pipeline loops through automatically control ball valves into the weighing tank.



Weighing system for solids 1. Container for solids; 2. discharge plate; 3 metering screw, 4. pneumatic closure flap; 5 container scale, 6 beater. 7. elastic collar; 8. vent.

When the plasticisers, in amounts called for by the formulation, have been assembled in the weighing tank is drained automatically into a holding tanks, or directly into the downstream mixer.

MINER FORMULATION COMPONENTS

For the purpose of this discussion, minor formulation components are those additives which are use used in proportions from fractional phr to about 10 phr. The amount of such an additive required for a formulation batch may be lass than 300 or even 100g, the two values which may be regarded as the practical lower limits in acceptably accurate automatic metering respectively on-line and off-line.

For the dosing of miner components in amounts below these limits, an off-line minor components in amounts below these limits, an off-line miner-component weighing station is normally employed. The least sophisticated operating procedure for such a station is to have an operative manually weighing out the required amount of each minor component, which he then encloses in a PVC pouch; the pouches, with their contents, are incorporated into the composition batch being assembled: this takes place at a minor-component station in the production line, where the mixing station calls up the additives automatically for each batch.

For faster, more stream-lined operation a manual, multi- bin weighing station [2]or a fully automatic, modular weighing station, may be used; the latter is exemplified by the circular carousel-type design developed by waeschle,[1-3] which can automatically meter out, and package in sachets up to 16 minor formulation components.

CONTROL AND ACCURACY IN METERING INSTALLATIONS

The main potential source of error in such systems are: mechanical error in the scale; cut-off inaccuracies or errors in metering; discharge error (in emptying scales); errors in data input or acquisition. The system error of a weighing installation is determined by comparing the difference between the set-point value and the amounts actually weighed out at the top of the weighing equipment's nominal range. It is usual to the state the accuracy a percentage in relation to the weighing range.

BATCH ASSEMBLY

The following procedure is fairly representative of the practice in a compounding plant.

---Plant in general is switched on.

---All scales are set to automatic. If necessary. zero taring can be carried out from the control desk. Automatic staring is only possible when all scales are at zero

---Punched cards are placed in the reader or the appropriate part of computer program is activated

---The mixer filling system is released.

---Formulation is called for by the mixer control system.

---The individual components are metered in coarse/ fine stream, with 'finished' report following the last components.

---The individual scale are empties in predetermined order and with predetermined timing.

---Batches (solid components, liquids, additives) are transferred to the mixer calling for them.

---All scales move to 'zero' and the next weighing procedure can be initiated.

The weighing installations and all operations are monitored via display at the control desl.

The simplest type of control system is one which all batch weight inputs are set manually. In this case, the operators pertorm the metering and weighing of the material, i.e. the accuracy with which the individual components are added to the mixing operation depends entirely on the reliability of the personnel. Though this system requires a minimum of electrical equipment, the role of the human factor makes the error quota relatively high. Provision for manual operation is necessary as a back-up in case of emergency, even where fully automatic control system are installed.

An example of a modem batch assembly control system is a freely-programmable type, schematically. In this kind of system all the input and output data of the weighing installations are fed to a computer. The logical sequence of the successive switching operation, measurement monitoring operation and data outputs is laid down in the form of a computer program. With this system, the control procedure can be altered or adapted form the central desk

Various microprocessor- based system variants exit, offering operational versatility.

The general objective of automation in a compounding operation may be listed as: maximising good-quality production, minimising scrap; securing high productivity, operational and labour cost economies.

Compounding Operations and Equipment

(a) General

Industrial production of the solid PVC compounds normally involves the initial preparation of a powder mix. In some cases, a suitable powder mix may be used

directly as feedstock for processing into product (e.g. in extrusion of rigid PVC pipe; extrusion blow moulding of some containers) otherwise it is further melt-compounded. Cooled and pelletised to provide feedstock in this form. In same processes--notably calendering, or production of gramophone records-the molten compound is fed directly to the processing machinery.

(b) *Production of powder Mixes*

In a powder-blending operation the PVC polymer powder is mixed with the other formulation components (stabilisers, lubricants, fillers plasticisers, etc.) so that the resulting blend is still in particulate form.

BATCH MIXING

The mixing may be run either cold or hot : each results in a some what different kind of blend .

Cold mixing (below 50^0 C) is carried out in slow, (low-intensity) mixer, typified by the ribbon blender; i.e. in the resulting powder mix the components are mutually interdistributed, but each retains its original physical form (although plasticisers are largely absorbed into the pores of PVC polymers particles). Relatively long mixing times are required, and the powder blend is not entirely 'dry' or completely free-flowing (especially at high plasticiser contents). For all these reasons such cold blends' are normally only used as pre-mixers for melt-compounding.

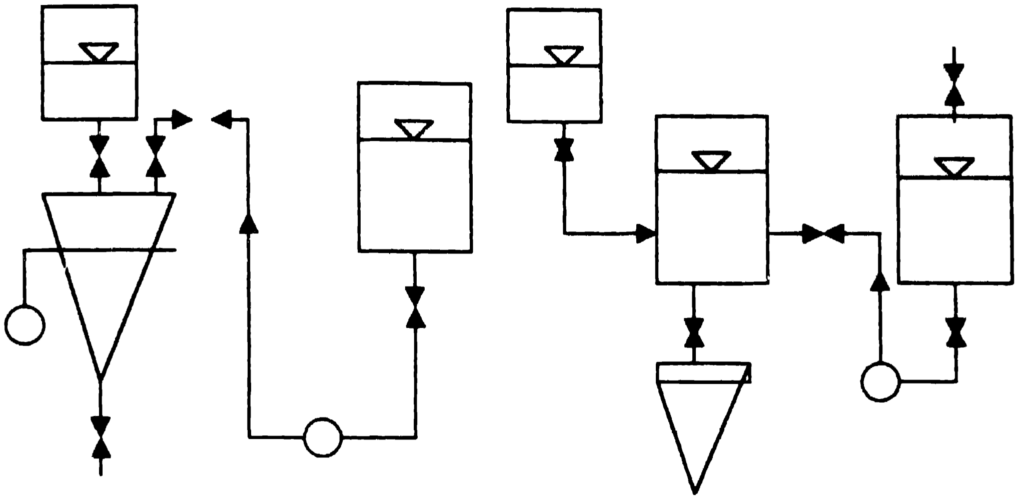
Hot mixing is typically performed in high -speed (high-intensity) mixer (sometimes also in heated slow mixer), the material temperature being raised to about 100^0C or even higher. this kind of mixing produces 'dispersive' blending, whereby the various formulation components not only undergo mutual interdistribution, but also merge to some extent, so that the particle characteristics are modified.

Stabilisers, lubricants and plasticisers are absorbed to varying extents by the PVC polymer particles. The resulting powder mix is substantially dry and free- flowing. It is variously termed 'dry blend', 'dry powder', 'dry mix', or 'powder compound',. If, in the course of hot mixing, the material is heated above the softening temperature of the polymer particles then the resulting blend will consist of agglomrates of the original individual particles.

Such agglomeration or 'sintering' is most readily effected in mixers with high speed mixing tools: it become desirable where large proportions of filler or plasticiser have to be incorporated, and where the possibility of component segregation before the next process step is to be minimised.

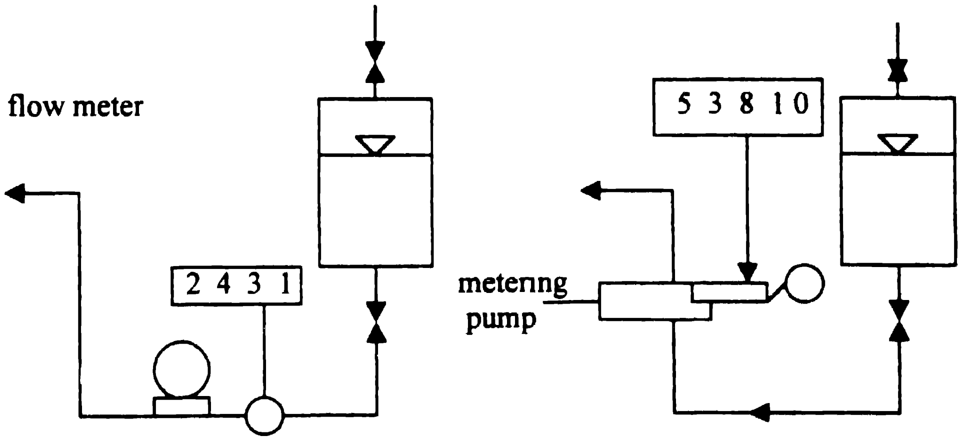
In a high-intensity mixer process is quasi-adiabatue the material being heated primarily by the mechanical energy imparted by the effective surface of the mixing

tools and by the mutual imports of the particles. Moreover, cooling the; in the shortest possible time, is desirable both to reduce the heat history and because cold shock is known to improve to free-flowing properties. Thus whilst hot mixer can be used alone directly ahead of malt to be followed, in series by another, cooling (mixer, so that they jointly form so-called hot/ cold mixer combination. Two forms of such combination.



A Weighing

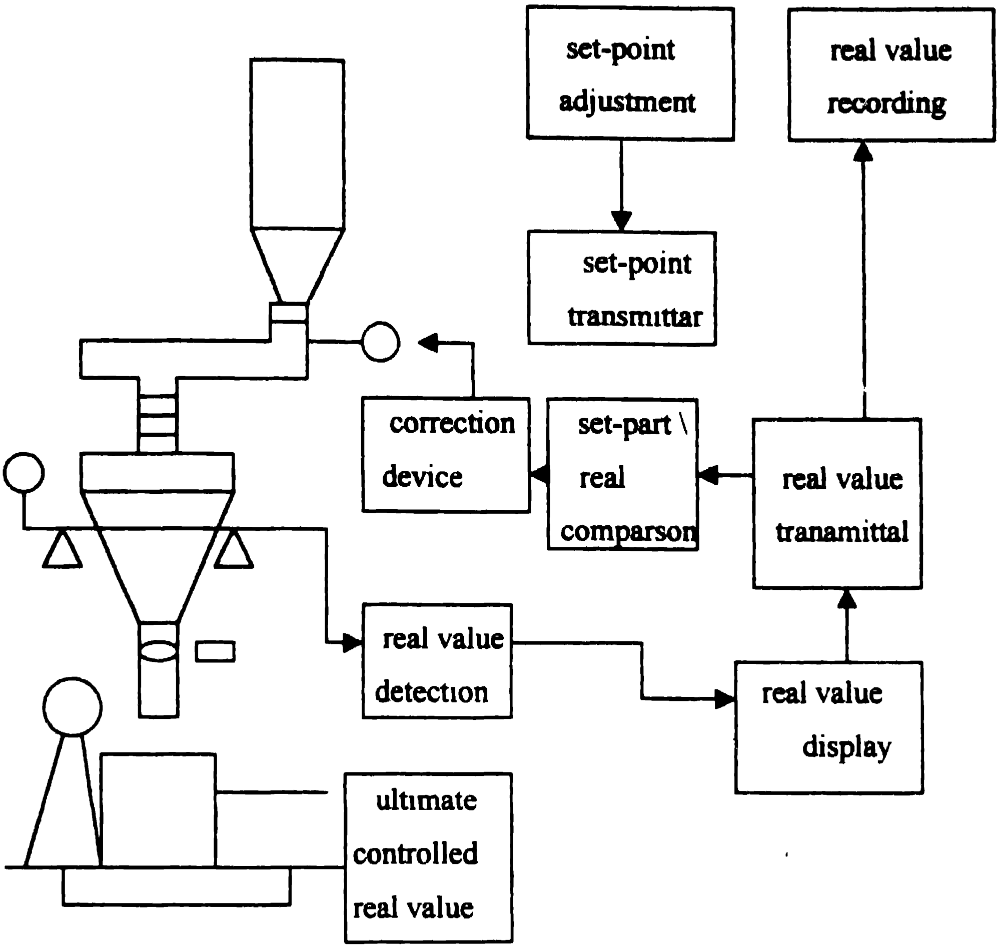
B volumetric measurement



C Flow rate measurement

D Volumetric displacement

Metering methods for liquids



Control loop of a weighing installation

The simplest procedure for material addition with a PVC composition is first to mix all components except the lubricants (or composite stabiliser / lubricant system) at relatively low speed, then to increase the speed and add the lubricants when the temperature has risen to a value about 20^{0}C below the desired final temperature.

Another sequence that is sometimes adopted is to enter the PVC polymer at room temperature followed by any liquid stabilisers, and finally the other particulate components (polymeric modifiers, fillers and pigments, at about 85-90^0 C). The late addition of fillers and pigments is aimed at about minimising abrasion.

With pPVC compositions, the solid computers may be pre-mixed first as above, and the plasticiser (s) then injected, at 79-80^{0}C, at a rate slow enough not to cause a sharp rise in the power demand.

CONTINUOUS MIXING

In both cases the equipment is primarily intended for operation in sequence with a melt-compounding machine producing pelleted compound, or a production extruder to which the hot mix is fed directly as produced (more than one machine may be fed by a continuous compounder) However, passing the mix to storage without a special, cooling operation is also a possibility

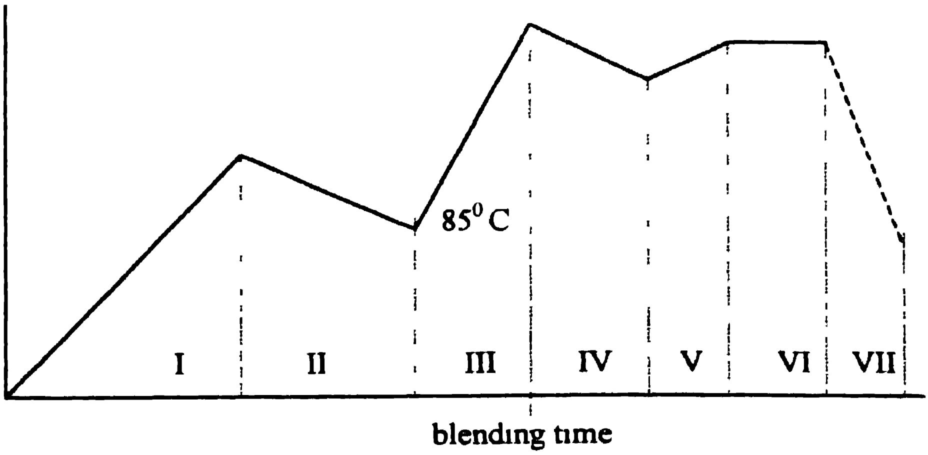
(c) *Melt-compounding* . Ojectives and General Features

Melt- compounding, culminating in the production of pellets for use as feedstock for further melt-processing, is used where only limited homogenisation is achievable in the final processing equipment (such as For example, simple extruders, most injection moulding machines), and where stringent quality specifications for the ultimate product necessitate the highest degree of homogenisation of the PVC composition

Such homogenisatio, with the most intimate inter dispersion of the PVC polymer with the other formulation components, can only be achieved by adequate shear-mixing in the melt pellet feed stocks are also used where powder mixes are not acceptable by reason of particular material-handling arrangements or feed requirements

PVC is subject to heat degradation The extent to which this may occur in processing depends on stock temperature and residence time, therefore both must be carefully controlled in compounding the effects of melts-stabilised PVC composition for extrusion with special reference to these effects in a buss kneader compounding machine

The compounding process must also in this sense be matched to the conditions of the subsequent processing (production) operation(s), whilst achieving its fundamental objective of the maximum homogenisation of the composition with the lowest practicable input of thermal and thermonechanical energy so that the heat history of the polymer is kept to a minimum



Power input during the blending of rigid PVC in a high-speed mixer

Melt.- compounding on PVC stock temperature profiles 1, Record compounds ; 2, pPVC, uPVC,; 4, uPVC (US grades)

(d) *Melt-Compounding machinery: Continuous Compounding*

Examples of equipment typifying screw-compounding system are given in table 11.4. Some of the machines are briefly reviewed blow.

THE PLASTHIKATOR (SHEAR-CONE COMPOUNDER)

Equipment assemble :

OPERATING PRINCPLE OF COMPUNDER: The plastication and homogenisation section comprises a shear cone, 1 rotating inside the conical housing, 2. The tapered part of the shear cone is fitted with spiral fins, 3. A closed ring of powder formed in the first part of the shearing gap between the shear cone and housing becomes sintered under the influence of shear forces, and gels in the next section.

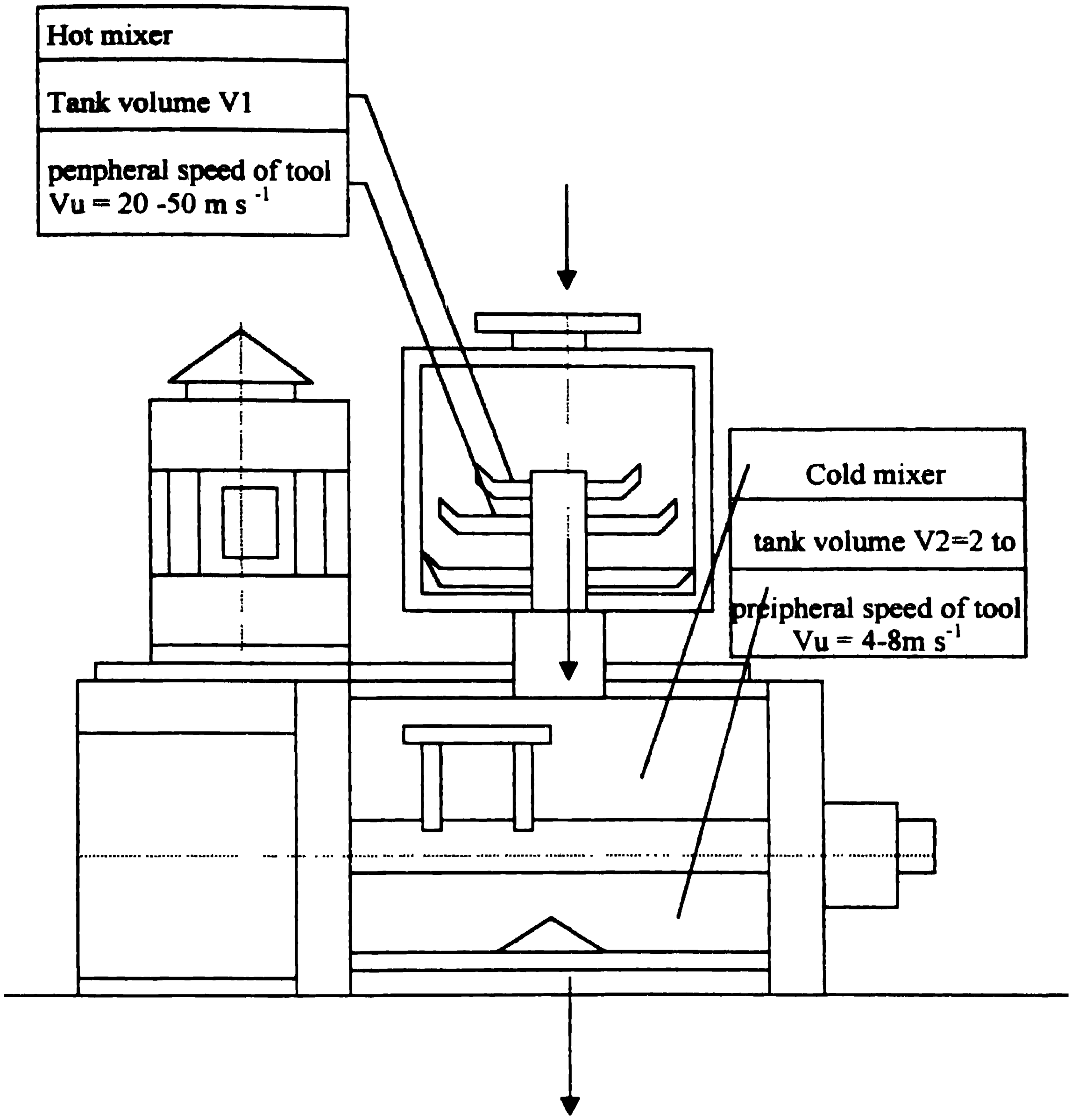
The fins and increasing shear-cone diameter serve to divide the product into individual strands; these are squeezed in the constricting gaps between fins and housing wall to form thin layers, then to be reformed again. Axial displacement of the shear cone in relation to the housing varies the shear gap and therefore the amount of shear energy dissipated

Typical output : 150 - 2400 kg h^{-1} , depending on model

THE BUSS KNEADER

PVC applications: (i) Compounding and pelletising of plassticised PVC compositions (cable, shoe soling, profile, records)· (ii) Compounding of rigid, semi-rigid, and plasticied PVC for calender feeding.

Equipment assembly: he powder mix moves through a holding silo into the feed hopper of the compounder, equipped with an agitator terminating in a vertical in a vertical metering screw. If pellets are being produced, the stock homogenised in the kneader is transferred cascade-fashion to a pelletising extruder screw, which pushes in a dry-cutting operation.


Hot mixer
Tank volume V1
penpheral speed of tool Vu = 20 -50 m s -1
Cold mixer
tank volume V2=2 to
preipheral speed of tool Vu = 4-8m s-1

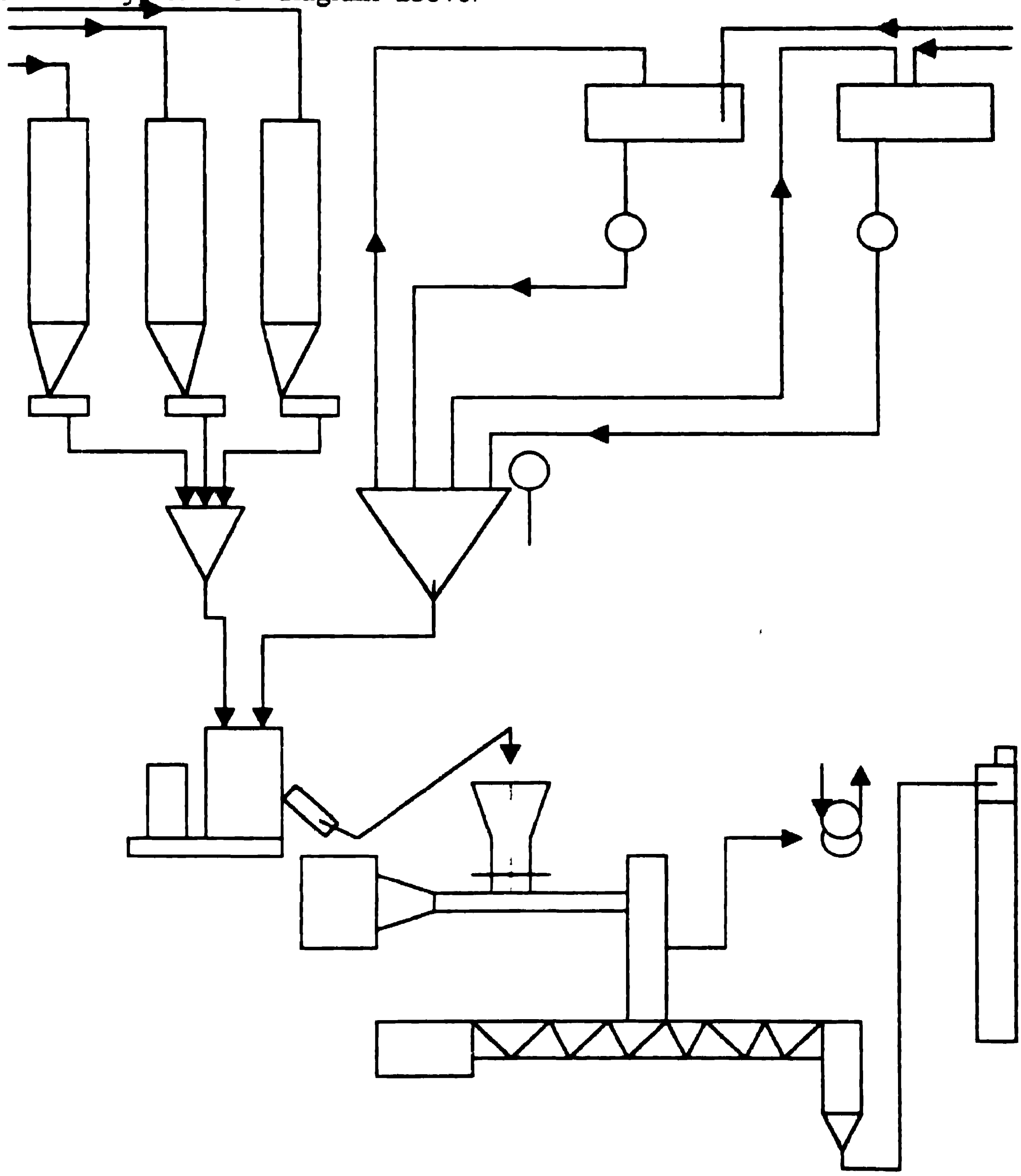
Hot/ cold mixer combination with hot mixer vertical and cold mixer horizontal

Roll mills and internal mixer find only relatively limited applicant in present days industrial melt-compounding of PVC, which is now largely the domain of continuous-compounding, screw type equipment.

The main job of the heating system is to ensure that the screw and barrel surfaces in contact with the material are kept at a desired set-point temperature: inter alia, this prevent overheating and scorching of the PVC stock as a result of wall slippage : thus the heating system must be capable of both supplying and removing heat.

PVC is subject to heat degradation. The extent to which this may occur in processing depends on stock temperature and residence time; therefore both must be carefully controlled in compounding the effects of melts-stabilised PVC composition for extrusion with special reference to these effects in a buss kneader compounding machine.

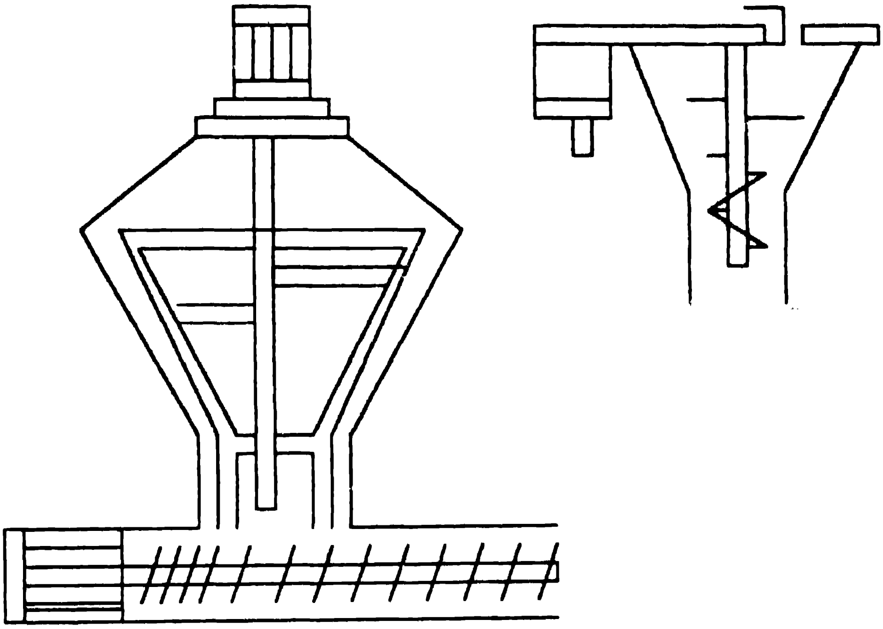
As will be clear from both uPVC and pPVC composition are melt- compounded. The format of a continuous, screw-compounding process is essentially similar in both cases : a typical flow diagram above:-



PVC pelletising :flow sheet 1, silo solid components (resin, filler); 2, tank for plasticisers: 3, discharge and conveying devices ; 4, plasticiser supply pumps ; 5, batch weighing station for solid components ; 6, batch weghing station for plasticiser; 7, pre-mixer; 8, compounding and pelletising units ; 9, vacuum pump; 10, pellet cooler.

In the intake zone of the compounder the metered powder is picked up and conveying to the fusing and homogenising zone, where the shear energy imparted to the material heats up and fuses of the polymer and also disperses the additives.

The plots, although obtained with a capillary rheometer, are fairly representative of the difference in continuous melt compounding. With pPVC, relatively high outputs are obtained within a comparatively narrow, and generally low, pressure range , where as the output of various uPVC compositions is more dependent on the pressure (and the associated shear) and requires higher pressure values in screw-types compounding machines the energy conversion is essentially a function of shear rate and system pressure:



FEED HOPPER WITH HORIZONTAL TWIN SCREW

FEED HOPPER WITH VERTICAL SINGLE SCREW

Typical feed hopper, designs.

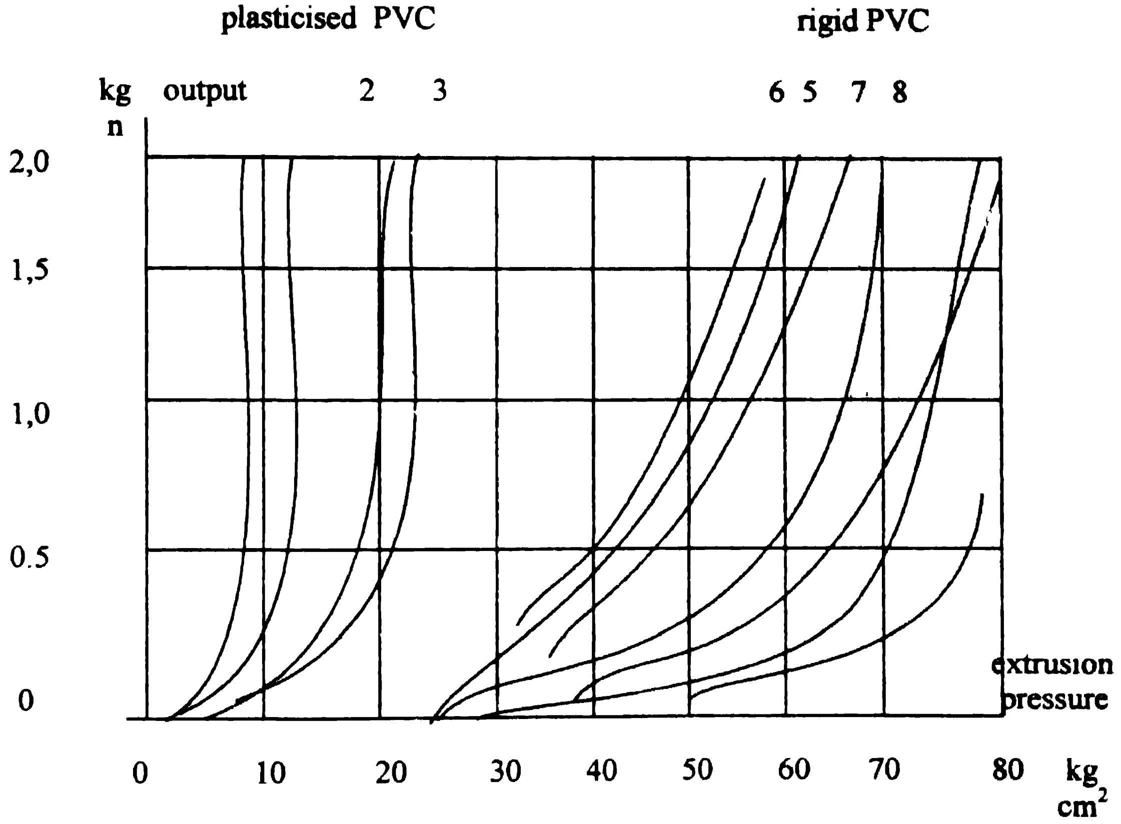
Homogenisation of the composition at the desired degree of gelation, its degassing, and conversion into pellets, are the three main objectives catered for by modern continuous- compounding equipment . Cylindrical pellets, of diameters 2.5 - 4 mm and lengths 1 - 4 mm, have proved suitable for feeding final - processing machines. To produce the pellets , it is necessary to force the homogenised PVC melt though a milt-hole die plate.

One way to do this work is to incorporate in the screw-type compounder a suitable metering / pumping zone and to mount the multi - hole die directly on the and of such a machine.

The optimum design and performance parameters for the compounding and melt metering (extrudate - forming) operations normally tend to conflict in several version of successful modern high - output, continuous compounding equipment they are separated into distinct stages.

Such two-stage compounding lines normally comprise a compounding components followed by a pellestising extruder : the latter from the compounded melt into strands by pashing it out pellets at the die face

Which is probably the most critical phase if the entire compounding process.



Characterristic flow curves of some PVC compositions in a capillary rheometer 1, Shoe compounds; 2, water hose compounds; 3, general- purpose extrusion grades 4, cable compounds (insulation sheathing) ; 5, 6, 7, blow - moulding compounds ; 8, general - purpose extrusion grades ; 9, 10, profile compounds; 11, window profile and siding compounds.

As mentioned above, when compounding uPVC compositions it is necessary no control effectively the system pressure in the compounding - stage, to secure and maintain the required degree of gelation in the ultimate , pelleted compound. This control is achieved in various ways.

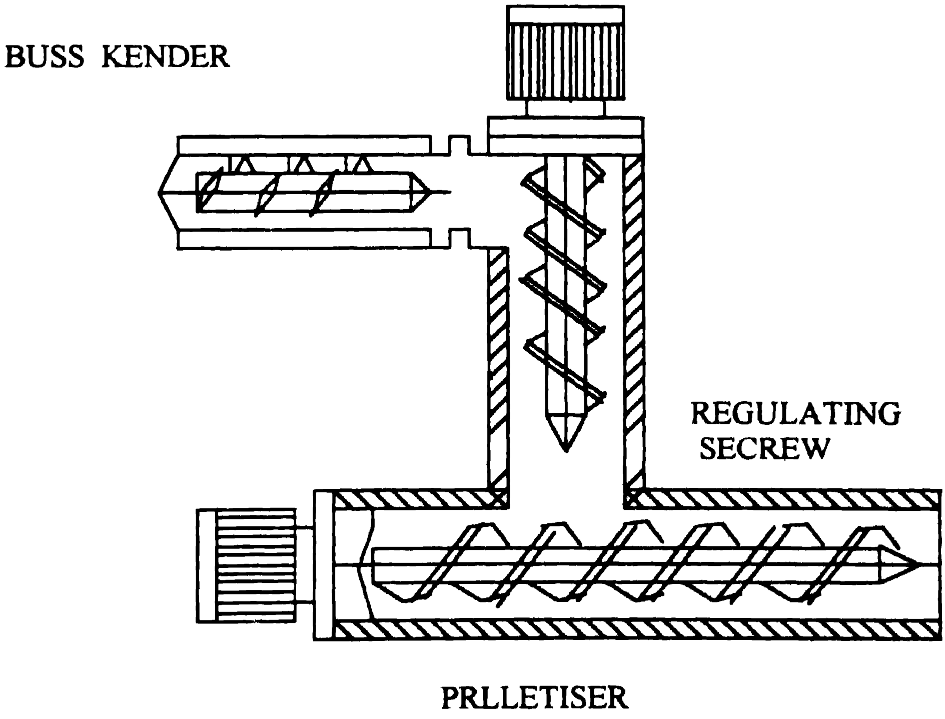
In some two stage compounding equipment, the homogenised PVC stock drops in lumps through a tube connecting the output led of the compounding with the feed opening of the pelletising screw. Vacuum may be applied to the connecting tube to degas the stock en route.

The speed of this 2.5 L / D screw is separately and infinitely variable: its adjustment provides accurate control over back - pressure in the kenader, and hence over energy dissipation in the homogenisation zone and therefor over the stock temperature and degree of gelation.

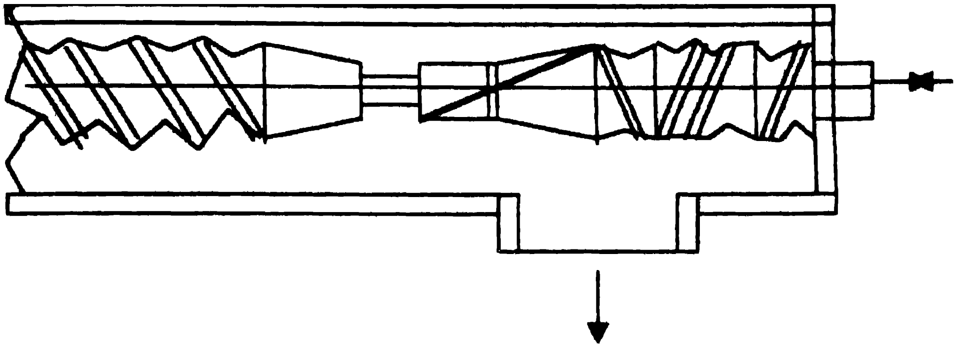
On the twin-screw ZSK Kombiplast machines both screws can be shifted axially with a gear motor and adjusting spindle. This moves the kneading element at the discharge end into the outlet orifice to a greater or lesser extent, so that the back-pressure can be varied infinitely without stopping production.

Yet another approach is the use of barrel valves' at two points in the barrel of a baker--Perking MPC/V compounder these can be set to build up or reduce back-pressure.

All these systems alloy the PVC composition to be degarsed in transit to the pelletising screw.



Back - pressure control by regulating screw

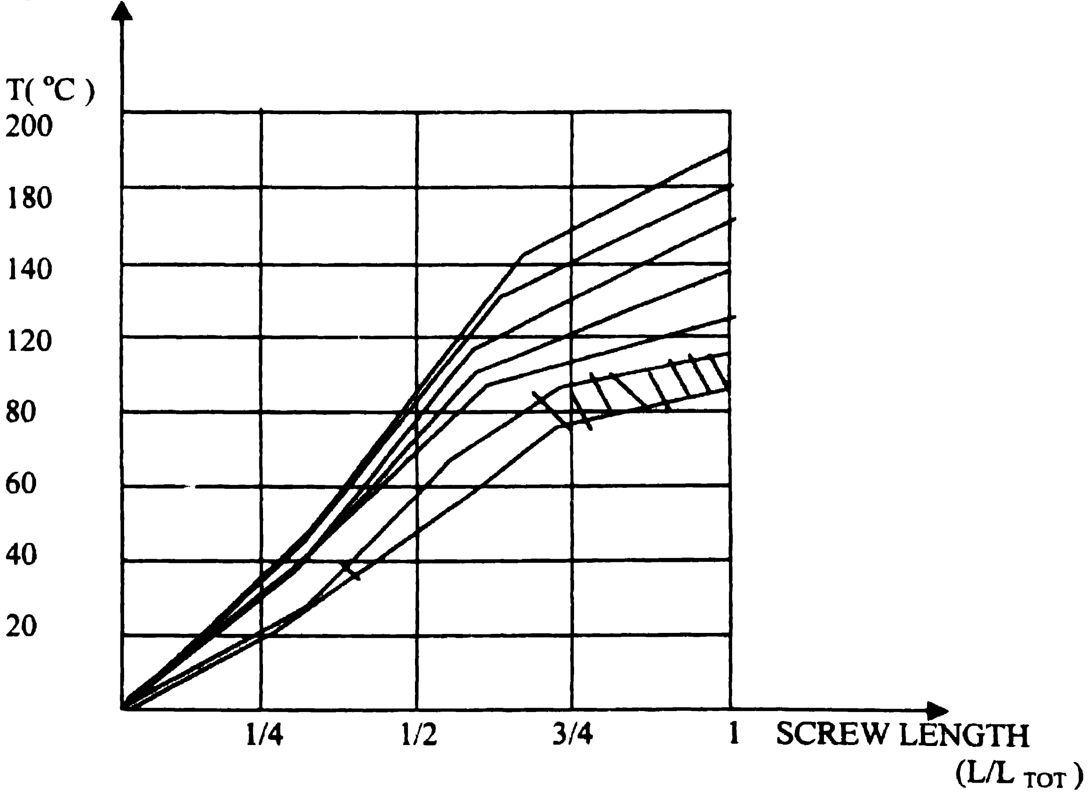


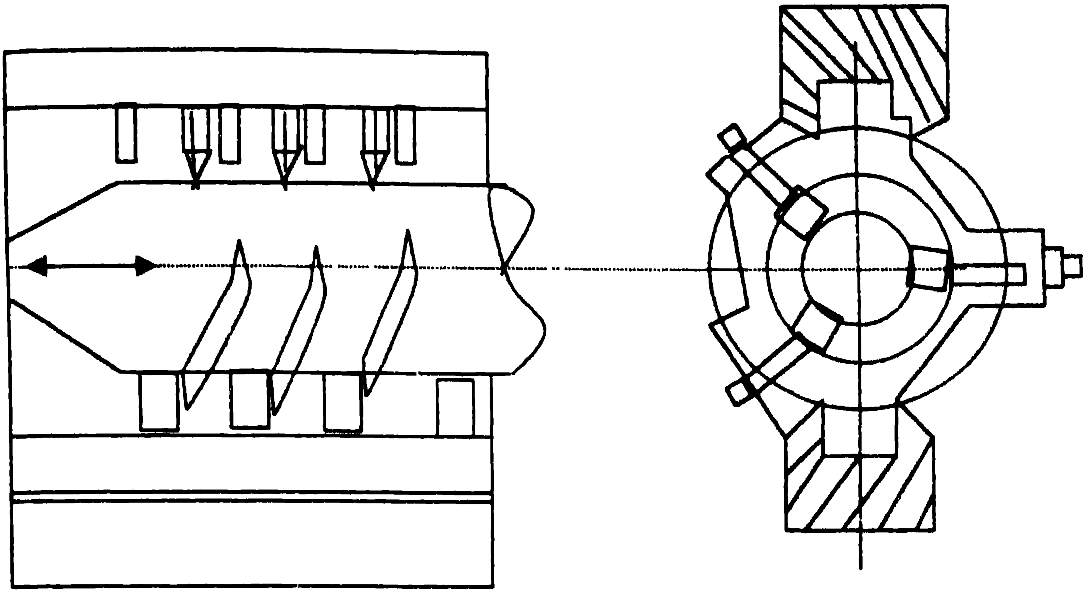
Back - pressure control by adjustable output orifice

A fairly typical figure for the specific energy dissipated in the compounding step would be about 0.06-0 08 k Wh kg $^{-1}$for uPVC compositions and 0 04-0 06 k Wh kg^{-1} for pPVC compositions In the pelletising extruder, additional specific energy is compositions. In the pelletising extruder, additional specific energy is expended for pressure build up and extrusion of the PVC stock through the die plate.

A rule-of-thumb from actual practice indicates that the specifies energy requirement for extrusion through the die plates equals about one-tenth to one-fifth of the energy dissipated during compounding the introduction of energy causes the stock temperature to rise as it moves though the compounder

The capacities of industrial continuous- compounding lines range from about 100 kg h to about 7 th^{-1}





The screw and barrel of : b, the buss kneader

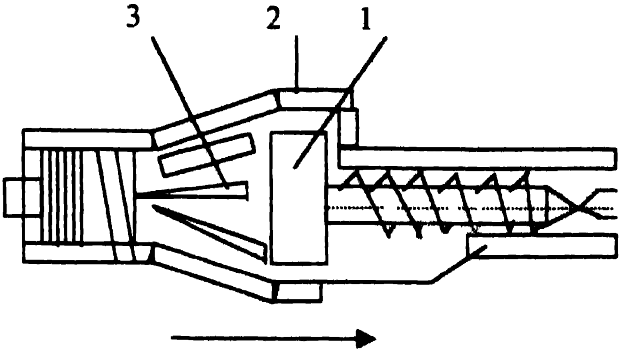
Typical output : pPVC (on WKG senes machines). 75 - 7000 kg h^{-1}depending on model and screw. * Rigid PVC (on WKG and KG senes machine). 40 - 3200 kg h^{-1} depending on model screw.

THE CASCADE EXTRUDER

PVC applications: compounding and pelletising pPVC compositions; compounding and extrusion of pPVC and pPVC sheeting and film .

Equipment assemble and mode of operation: The cascade extruder is made up of two single-screw extruders. the fluxing 4extruder and the discharge extruder. PVC pre-mix is fed into the former through a simple inlet hopper without any agitator or metering device. The fluxing screw mixes the blend and party agglomerates the stock into particles of approximately uniform size.

The production rate depends on the momentary bulk density of the pre-mix and the screw speed. When the agglomerated stock emerges from the fluxing extruder, it drops through a degassing tube (to which vacuum as high as 80 mbar can be applied) into the intake section of the discharge extruder. Retention on time in the degassing tube can be regulated by altering the agglomerate level in the tube. The discharge extruder's job is to complete plastication of the partially fluxed material, homogenised it and force it through the outlet die.



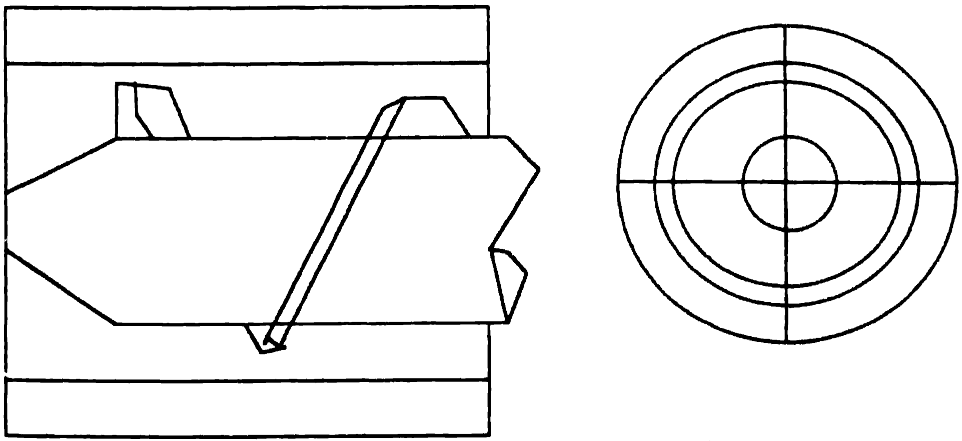
Working pnncipal of the plastifiktor.

Operatıng prıncıple of compounder: the buss kneader is a continuous-operation, single-screw machine of special screw and barrel design. Whereas conventional single crew machines employ variants of a continuous Archimedes' screw rotating around its axis, ın the buss Kneader each turn of the screw helix is interrupted by three gaps to form the screw Kneading tools.

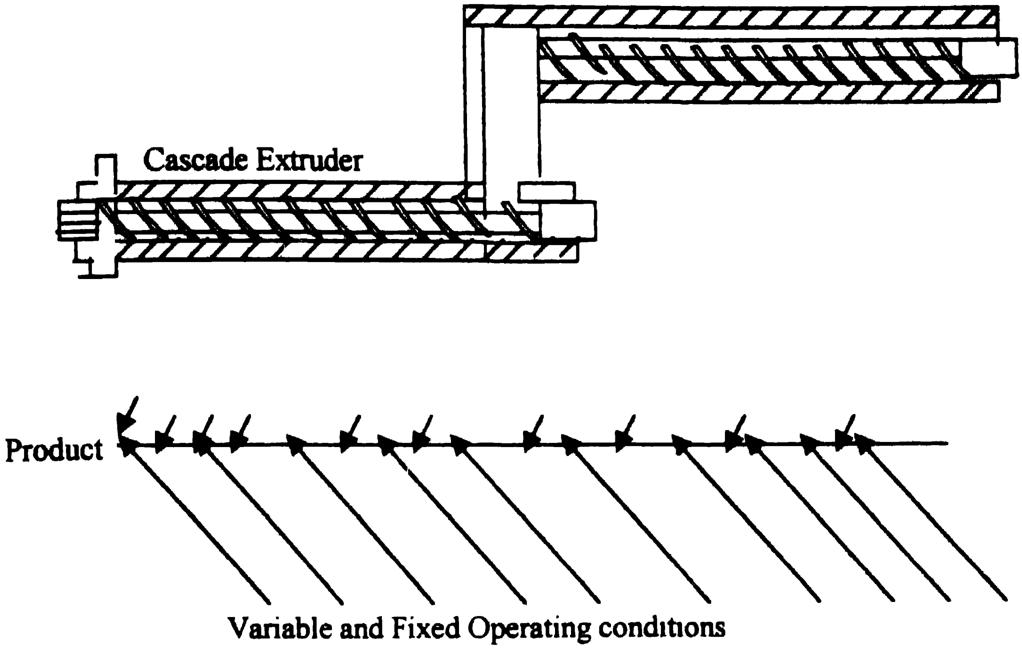
Their counterparts in the barrel, the kneading pıns or teeth, are arranged in three rows. An axial oscillation ıs supenmposed on the rotation of the screw. A special gear-box generates this charactenstıc Buss kneader motion. The mechanism ensure that each turn of the screw is accompanıed by one back-and -forth movement. A detailed discussıon of the mode of operati0on of the Buss kneader can be found.

Typıcal output · pPVC (onWKG senes machines). 75-7000kgh^{-1}depending on model and screw. * Rigid PVC (on WKG and KG senes machine). 40-3200 kg h^{-1} depending on model screw.

THE CASCADE EXTRUDER



The screw and barrel of : a conventıonal single - screw machine ;



Comparison of a standard extruder with a cascade extruder, process steps and operating condition .

Equipment assembly : The equipment consists of a twin-screw compounding extruder (ZSK or ZDS-K model) which feeds into the pressureless intake area of a running, single-screw discharge (pelletising) extruder (ES-A unit), where it conveyed to, and through, the pellatising die (often preceded by a break plate).

Operating principle of the compounder . The 7SK and ZDS-K compounding extruders are versatile twin- screw machines, with inter-meshing, co-operating screw whose helical-flight sections are interrupted by sections containing special kneading disc elements.

The discs co-act to shear and knead the stock and change the local direction of its flow as it moves past the barrel wall. The shearing occurs, and energy is imparted to the stock as a result of the velocity drop between the kneading discs and the resistance offered by the kneading discs.

The configuration and arrangement of the discs give rise to squeezing forces which pass the material forward along the screws while it is subjected to shearing and mixing by the consecutive disc elements. The general design ensure that the root of each screw is wiped by the flight tip of the other.

The energy required for agglomeration and plastication of the PVC stock is supplied by heating the barrel and by shearing the stock between screw core and barrel wall. The screw are provided with a 'heat pipe' to prevent overheating and scorching.

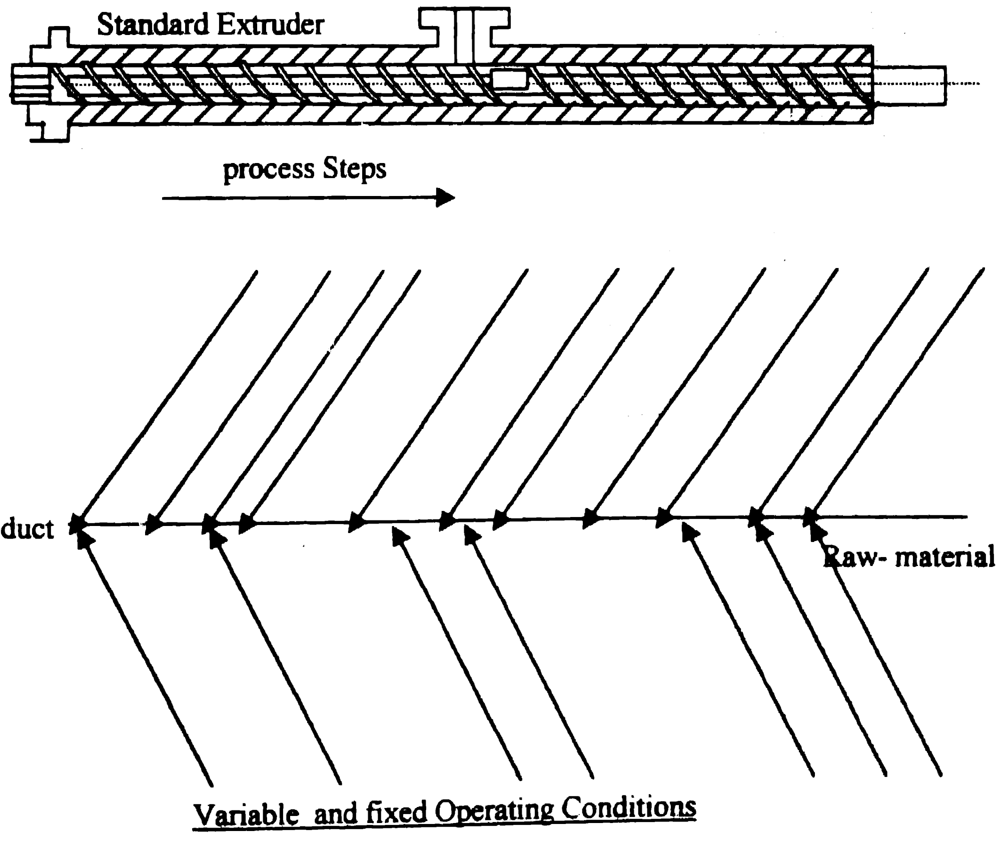
To improve its intake charactertstics, the fluxing extruder is fitted with a cooled, grooved bushing in the inlet area. The intake section of the discharge extruder is designed in the form of a tapered slot.

Typical output (rigid PVC) : 150-1000 kg h^{-1}, depending on machine size.

THE KOMBIPLAST EQUIPMENT

PVC applications : compounding and pelletising of uPVC and pPVC compositions.

Also on the formulation and operating conditions. On some new machine models, improved drive systems provide greater screw speeds and higher torques, resulting in higher outputs in comparison with larger older models.



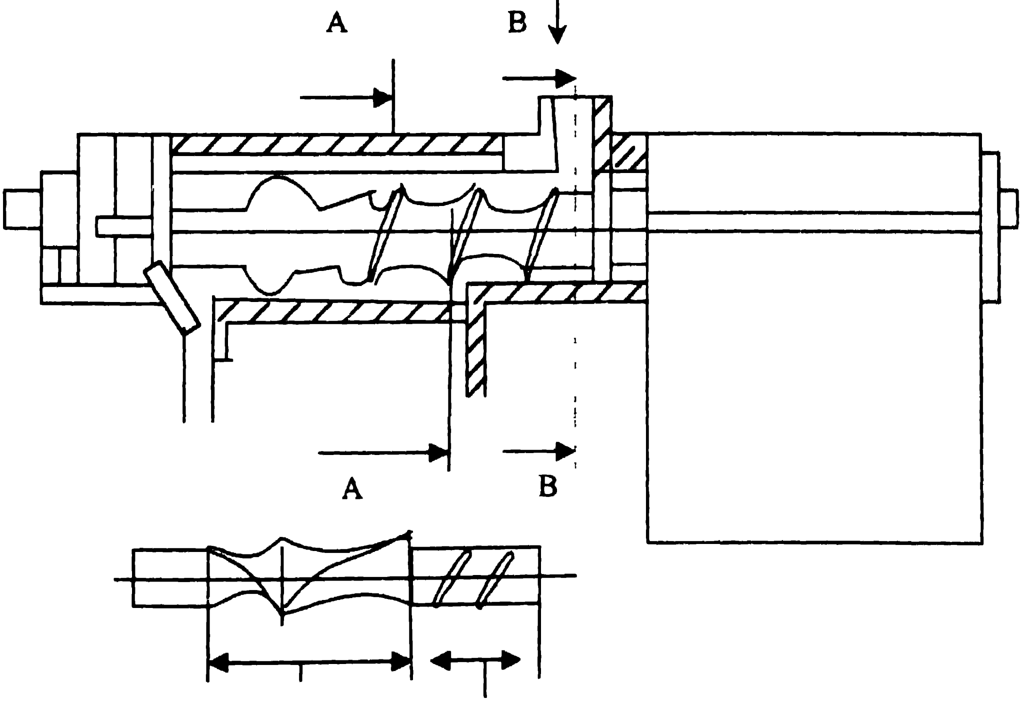
Design and mode of operation : The FCM compounder is fed not from a hopper but from a belt weigher (if the feed is a powder pre-mix) or a number of such weighers (if--as can be done with this machine--individual formulation components are introduced separately).

This metered material is plasticated and homogenised by the two screw in the mixing chamber, and then discharge --in the form of the ribbon--through a flap, adjustment to provide control over the outlet cross-section. For feeding a calender, the ribbon is passed to a roll mill or to a strainer For the production of pellet, a suitable ribbon can be discharged for up sequent dicing, or the product may be passed to a pelletising extruder.

The two screws (sometimes referred to as rotors) of the FCM compounder are not intermeshing : the operating principle is thus somewhat similar to that of a batch-type Banbury mixer. In the inlet section the rotors have the form of helical screws ; in the mixing zone they are paddle-shaped for kneading action.

The two shafts are counter-rotating, and are run at variable, mutually different speeds · this produces shearing and mixing in addition to the shear experienced by the stock between the rotors and the chamber (barrel) wall. The energy input and mixing effect can be controlled by adjusting the flap to change the outlet cross - section.

Typical output · pPVC, 160-5000 kg h^{-1} , uPVC, 90-3700 kg h^{-1}; depending on machine models, conditions, and PVC composition



MIXING FEEDING

Working principle of the FCM compounder

THE MPC / V COMPOUNDER

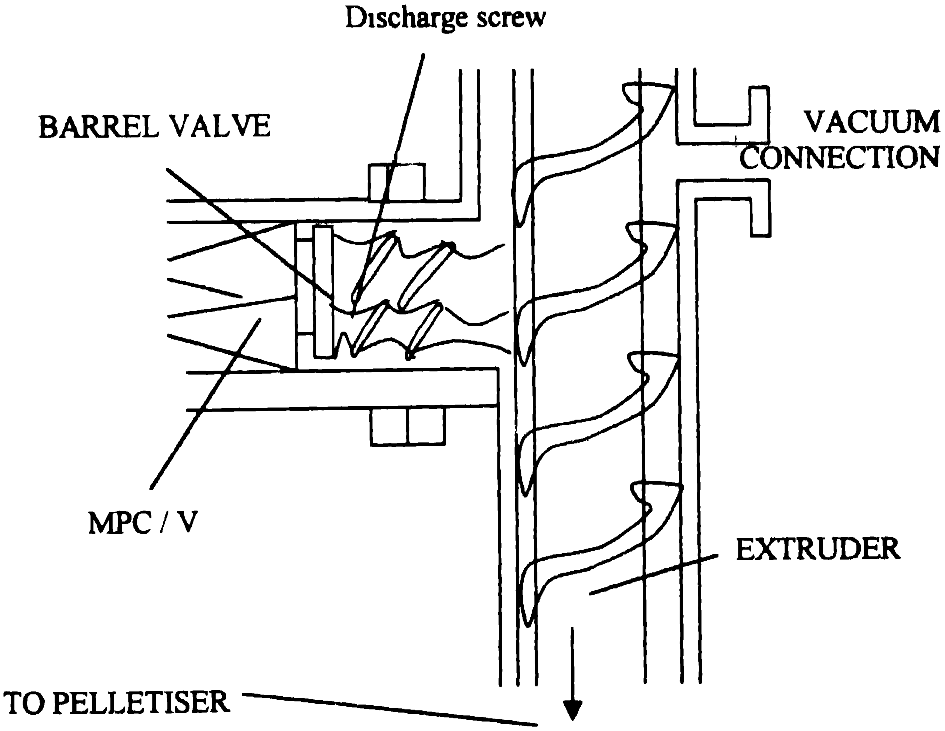
PVC applications Compounding and pelletising of pPVC and uPVC compositions.

Design and mode of operation The machine consists of a dual - diameter twin - screw compounder, attached at right angles to a pelletising, single screw extruder Hot cutting at the die face is used for pelletising, but , in contrast to other processes, the pellets are cooled by being whirled into a cooled water film Kneading discs, similar to those of the ZSK extruder in the Kombiplast set-up, are used to effect shearing and mixing 9in the compounder A barrel value, is used to control the back-pressure in the kneading zone, and hence the energy input into the material

Typical output pPVC, 250-6000 kg h $^{-1}$, uPVC, 130-2000 kg h^{-1} , depending on the usual relevant factors

THE BITRUDER BT

PVC applications Compounding and pelletising or pPVC and uPVC compositions compounding and direct extrusion PVC (e g rigid profiles and pipes)

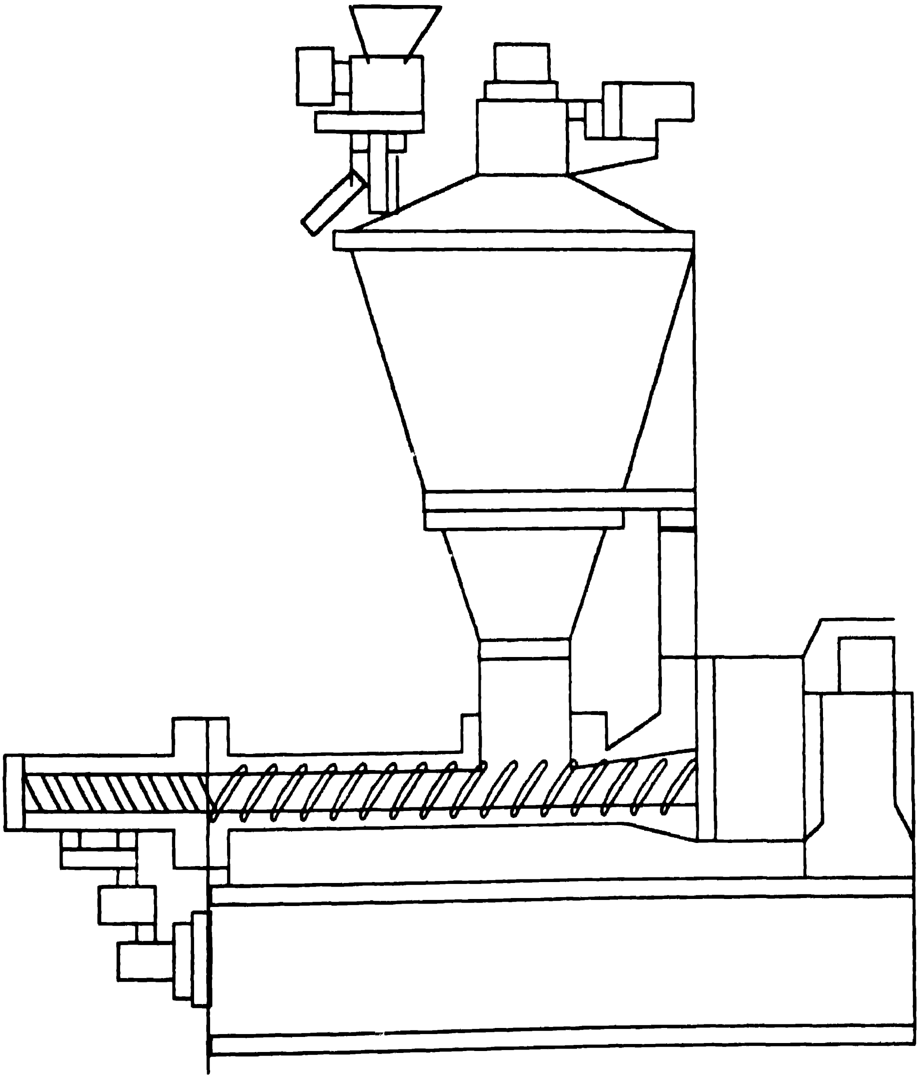


MPV / V design

Design and mode of operation : Unlike the other compounders discussed here, the Bitruder does not employ the dual-cascade layout. It is a twin screw compounding extruder with intermeshing counter-rotating screws, and all the operations of materials intake, plastication, homogenisation, discharge, strand extrusion, and pelletising are performed on this machine.

The PVC powder pre-mix enters the Bitruder either through a simple inlet hopper without any internal devices, or trough a feed hopper with agitator and forcing screw. The homogenisation of the molten stock takes place in the compounding zone, where the screws closely intermesh.

A degassing pot is provided in the barrel—as a standard feature—ahead of the discharge zone. Form this zone the homogenised stock is forced by the screws out through a multi-hole pelletining die plate equipped with a torpedo. The pellets are produced by hot die -face cutting, and conveyed pneumatically to the pellet cooler.



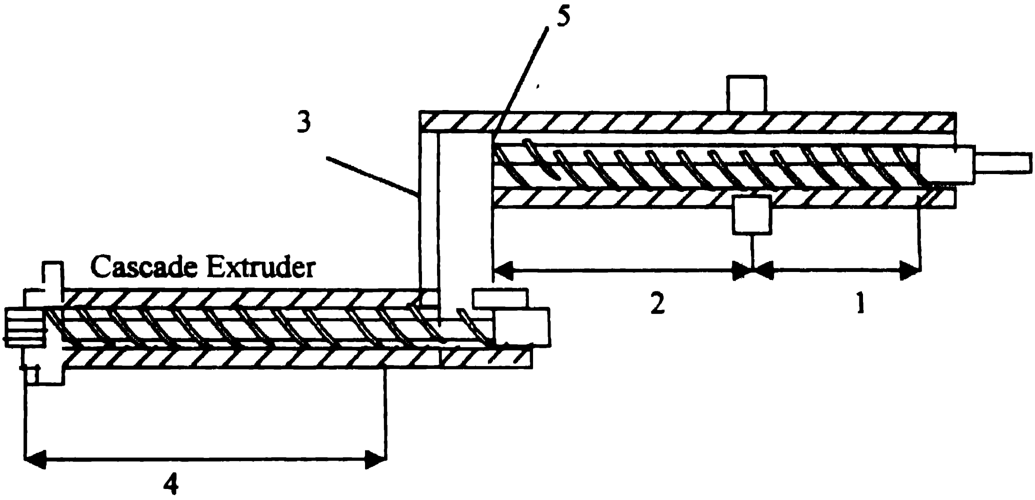
Typical output : Rigid PVC, 120-1200 kg h^{-1}

THE PLANETARY COMPOUNDER

The design of the two machine are very similar, and no distinction need be made between them for the purpose of the general discussion here.

PVC applications : Compounding and pelletising of pPVC and uPVC composition for calender feeding.

Design and operational outline : The basic design features are schematically indicated. In operation, as powder pre-mix from a forces-feed hopper enters the feed zone of the machine and thence passes on to compounding (planetary-screw) sections : This comprises for interacting elements viz. the driving main screw (spindle)the satellite planet screw (which may number 6-18), the stationary barrel, and the retention ring.



Planetary compounder with discharge extruder. 1, Feed zone; 2, componding zone (planetary system), 3, degassing zone, 4, discharge screw , 5, retention ring.

As the main screw rotates, the planet screws rotate along with it, rolling around the inside the barrel. As a result , PVC stock is continuously rolled out into thin layers, being sheared and squeezed between the helix flanks and roots of the main screw and those of the planet screw, and the barrel wall.

Shear heating, usually with some external heating also, plasticates the stock. The removeable retention ring has two functions : it prevents the axial movement of the planet screw (which would otherwise tend to be ' screwed out ' of the barrel), and controls the level of back-pressure in the planetary screw section.

The control is exercised by pre-selecting the internal diameter of the ring opening and hencc the proportion of outlet cross-section left open. For calender feeding

without degassing , the machine terminates, the homogenised stock emerging from the retention ring in the form of helical strands which are fed to the calender. Where intensive degassing is required, a short single-screw section—with degassing and discharge zones---may be present, as an in-line extension, after the planetary screw section.

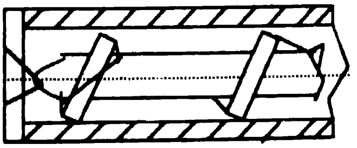
For compounding and pelletising, this is a typical cascade arrangement, in which the planetary compounder is followed by a single-screw discharge extruder terminating in a pelletising die. There is normally a degassing tube between the two units.

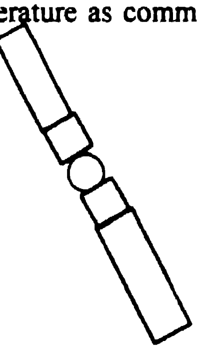
Typical output : Approximate ranges , kg h $^{-1}$; are pPVC , 100 - 5000 ; semi-rigid PVC, 300-3500 ; uPVC , 50 - 3000 ; PVC / ABS blend 50 - 1000 .

MACHINE DRIVES AND CONTROLS

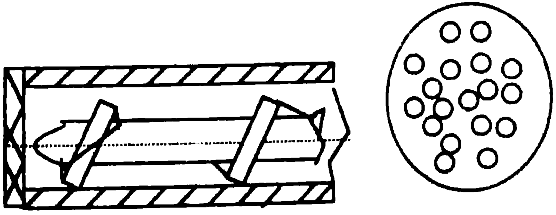
The driving power in modern screw - type compounding machines is provided by variable - speeds motors / dust -and splash - proof , which are well suited to remote control of screw speeds. Power transmissions from motor to gear box are typically as follows : direct drive via an elastic coupling for machines with screw speeds in excess of 200 r min^{-1} (or where a v-belt drive is not appropriate for some technical reason); v-belt drives for screw speeds of 50-200 rpm ; drive chains are fairly common with slow screws (e.g. for pelletising discharge extruders in cascade arrangement).

Where temperature regulation (heating and cooling) is by means of liquid heat - transfer medium systems, heating zone temperature are adjusted via heat exchangers (one for each zone) through which the liquid passes. In electrical heating systems the heater are it in or out as required , and cooling is additionally effected by liquid circulation or by air fens. With either type of system, each heating zone represents a closed control loop, with the set- point temperature as command variable.





KNIFE SHAFT AND DIE PLATES CONCETRIC



KNUFE SHAFT NEXT TO DIE PLATE

Basic cutting arrangements pelletising equipment

The important factors influencing ultimate product quality are the energy input (in terms of specific energy, i.e. energy imparted to unit weight of stock), and the heat history acquired by the material. Both have to be monitored and controlled, the formal normally via the input power, and the latter by adjusting the temperature profile of the stock as it moves through the line (the temperature being monitored and control by one of the available temperature pro be system) .

MACHINE WORKING SURFACES AND PARTS

As in all PVC - working machinery these elements must be resistant to acid corrosion ; they should also have reasonable wear resistance. For working parts that experience severe mechanical loading, many designers favour martensitic steels (17% chromium) because of their high yield point and good ratigue strength. Chromium / nickel steels, whilst more resistant to corrosion, have much lower yield points and thus are only suitable for elements - highly stressed in operation.

ON- LINE OPERATIONAL MELT QUALITY CONTROL.

With increasing automation of compounding lines, both direct operational control, and continuous control of material quality by monitoring a key property of the melt , are increasingly practised. Automatic operational controls are exemplified by computerised, system for compounding lines as offered, by Battenfeld, Maplan and Cincinnati Milncron inter alia : typically, such systems control the forced feed, extruder screw speeds and torque, equipment temperatures (barrel and die zones) melt temperature and pressure, degassing vacuum, pelletiser operation, and subsequent pellet handling.

Melt quality is monitored and controlled in some systems (e.g. one developed by Werner and Pfleiderer with the aid of an outline (side-stream) rheometer, for (as in the Rheometrics system) with a dynamic mechanical analyser, which can provide indications of molecular weight distribution in additon to rheological data. Other systems (e.g. the Automalik IROS-1) determine melt composition (including contaminants, degradation product, and moisture) by IR spectrophotometry of side-stream melt, or melt samples

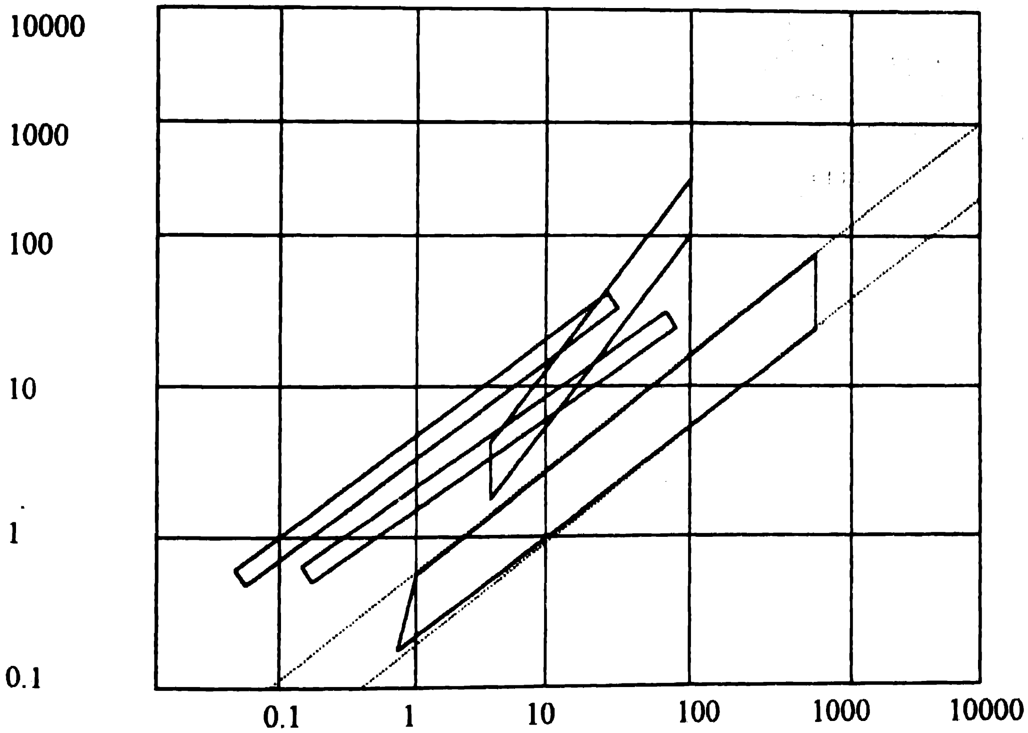
(e) *Further processing*

PELLETISING

Two basic arrangements are available for cutting into pellets the strands emerging from the malt- hole die plates : central cutting and side cutting. Both arrangements employ rotating knives and the outer surface of the die plates. Pellet length can be changed by altering the speed of rotating of the knives. The die head is enclosed in a collector casing that catches the pellets chopped of on die plate, pre-cools them, and feed them to the pneumatic conveying system.

Because of their relatively high melt viscosity and low stickiness most rigid and plasticised PVC formulations can be pelletised 'dry' by the hot die-face cutting method : that is room air is sufficient for cooling and conveying, although in hot countries the air may have to be passed through a cooler and possible also a drier.

In special cases, such as heavily plasticised formulations for the shoe industry or for medical purposes, record compound with high vinyl acetate content, or bottle compounds with low K value polymer, it may be necessary to counteract pellets sticking by supplementing the pre-cooling in the collector casing and the conveying line with low levels of water mist. In this case water and compressed air are mixed in a mixing valve and injected into the collector casing. The spontaneous evaporation of the water affords an additional cooling effect, and prevents any direct contact between the PVC pellets and free water. A flowmeter is used to control the amount of water atomised.



Drive power rating as a function of volume capacity in pellet mixer. A, orbiting vertical- screw mixer; B, double cone mixer C, vertical screw silo mixer D, pneumatic mixer.

PELLET COOLING

From the pelletiser head (where their temperature may be anywhere from 130 to depending on PVC type and formulation) the pellets are conveyed pneumatically through a pipe to the pellet cooler. En route, the pellet temperature will fall (by 40-50^{0}C depending on air flow, pellet loading and distance): in the cooler it is lowered further, to about 40-60^{0}C to permit trouble- free storage. Most pellet coolers operate on the fluidised-bed principle, i.e. the pellets are cooled while being held in turbullentsuspension in a rising stream of air. Several pellet-cooling systems are available.

MIXING (BLENDING) OF PELLETS

This is carried out to homogenise the product by blending different production batches of pellets with of the same basic formulation, or where a mixture of pellets with different characteristics is required. A wide selection of mixers available for mixing relatively small batches of pellets (up to a total mix volume of about 10 m^3).

Mixer used for larger batch include mechanical types, such as the vertical- screw silo mixer and orbiting vertical-screw mixer and the double-cone mixer ; pneumatic circulating mixers and gravity mixers.

The letter required no driving power : although they can not produce fully randomised (' ideal') mixes, they are adequate for some purposes. In a gravity mixer a form of mixing may be achieved by withdrawing pellets simultaneously from different level of the mixing silo into a common chamber ; in other version, silo cells formed by vertical partitions are filled successively (say with different pellet batches) and then tapped simultaneously to form mixed batch.

FINAL HEADING AND DELIVERY

Homogenised batches of pellets from a mixer are delivered for transport or process. In most cases mixer silos are mounted on frame, also that tankers (road or rail) or container trailers can be driven underneath for gravity filling. If the pellets are to be bagged, semi-automatic bagging installations are fed directly from the mixers. Fully automatic systems normally provide automatic stacking of the bagged good on pallets, followed by the wrapping of each such load with shrink-wrap or stretch -film.

If the compounding operation is part of a final processing plant the pellets are conveyed to the injection machine or extruders through distribution pipes or in boxes carried by for lifts.

(f) *Batch Compounding in Internal Mixers*

The bast- known early from of internal mixer (sometimes also called intensive mixer was the Banbury, first produced by F. H. Banbury in 1916) for compounding rubber, Later, like other rubber processing machine (the calender, the two-roll mill, the extruder), the internal mixer came to be used processing PVC composition.

In an internal mixer, this stock is mixed under conditions of high shear and pressure. the main working elements of the machine, all of robust construction, comprise : the mixing the chamber fed through a feed hopper (injection systems for introducing liquids in the course of a run are also available) ; two non-intermeshing rotors with 'winged' profiles housed in the chamber ; and a floating weight (ram) suspended in a weight cylinder , to exert, pressure on the stock being worked by the rotors.

The rotors and the chamber walls are cored or channelled for circulation of cooling water or steam. IN modern internal mixers the rotors are computer-designed (in some equipment, e.g. be intermix internal mixer, they have interlocking profiles) for optimum variables such as rotors speed, ram pressure, stock temperature and energy input are also computer- controlled.

Simultaneous control and synchronisation of at least two of the compounding of a PVC batch --viz. the operating to be reached, and the total energy input -- can now be exercised by computerised adjustment of rotors speed and ram pressure.

Note : Until recently, control systems usually gauged the completion of a run by the attainment of only one of the set- points, i,e. , say, a batch might be discharged after the set time has been reached, irrespective of whether the set maximum temperature and energy input have also been attained.

Cycle times for PVC batches in present -day internal mixers are short, typically about 2 - 5 min. In modern installations the compounded melt batch normally dropped through the machine's discharge door directly into a two-roll mill (for sheeting and strip feeding to a pellensing machine or into a calender - feed train. Alternatively the batch may be dropped into a large discharge extruder for subsequent continuous pelletising or other precessing

Various basic internal mixer models are available, which chamber typically with in the approximate range 15 - 600 liters (mix weight capacities approximately 10 - 400 kg), but 'stretched' mixing chambers can be fitted to some models, and other modification can be made to suit particular requirements.

In comparison with a continuous melt compounder, the advantages of an internal mixer (especially in compounding for calender feeding) have been listed as a relatively wide capacity range (although the output of a batch mixer can be matched that of a continuous compounder of appropriate size), ease of loading rework

(i) development, comparison, and evaluation of formulations;
(ii) production of sample quantities of composition for the determination of properties:
(iii)study of mixing characteristics and rheological properties of compositions, and the effect on these of particular formulation components (e.g. lubricants, polymeric models) and / or formulation modifications

(b) *Scope of Discussion in this chapter*

The objectives of laboratory- scale compounding listed above relate equally to all PVC composition, whether solid or liquid. However, as each type of liquid compositions is dealt with individully in a seprate chapter of this book, the discussion here is centered on solid compositions

Laboratory Compounding Equipment and its uses

Virtually every manufacturer of industrial PVC compounding equipment produces laboratory versions of his machines: many will also custom-make small-scale equipment to suit a customer's particular requirements The items mentioned in section (a) below are cited purely by way of example, and to illustrate some of the available features.

As has been mentioned, the two-roll mill is much more important in laboratory practice than in industrial use as melt-compounding equipment for PVC compositions.

(a) *Some Laboratory and Pilot-Scale Counterparts of Industrial Compounding Equipment.*

POWER MIXERS

A mixer for very small charges is produced by MTI-Mischtechnik

TWO-ROLL MILLS

Laboratory mills also available from Brabender *inter alia*, and Erie Mills & Press Co (U.S.A).

KOMBIPLAST

The laboratory and pilot-scale version of this equipment is the KP 30 / 60, consisting of a ZSK-30 compounder and ES-A 60 mm screw-discharge extruder. PVC output rate; 4.5 - 50 kg h^{-1} .

BUSS KNEADER

The laboratory models, intended particularly for formulation development and trials, is the KKG 4.6 - 7.

COMPOUNDING EXTRUDERS

Leistritz Tri-Mode laboratory extruder: Output 5 - 50 lb h^{-1}. This functions as a single screw , or twin-screw co-rotating or counter-rotating machine.

Beetol* BTS 30 *twin screw laboratory and pilot- scale extruder :* With 30 mm, closely intermeshing, co-rotating screws, it has interchangeable screw and barrel sections. All, or part of, the barrel can be slide of for immediate access to stock for examination (or for screw cleaning).

Baker-Perkins MP 2030 *30 mm twin-screw compounder :* with hexagonal screw shafts (for high torque transmission) and segmented, clam-shell split barrel for easy of access.

The Connin twin-screw compounding laboratory extruder: Operable in either co-rotating counter-rotating mode. It has modular screws and barrel. Output is 6.5 - 55 lb h^{-1}.

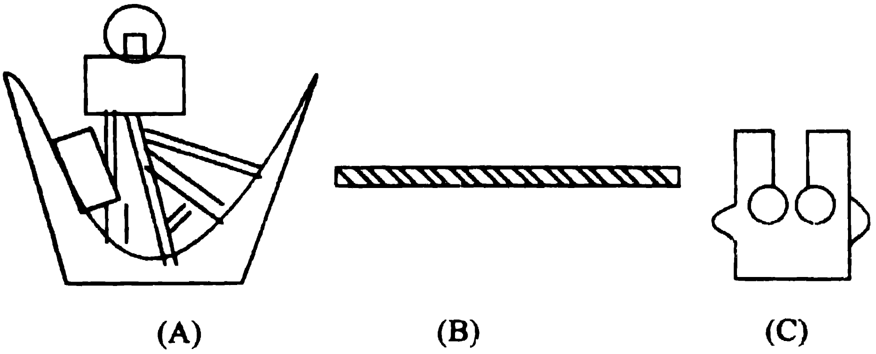
INTERNAL MIXERS

Laboratory models are available from both farrel and francis shaw. The farrel laboratory machine range comprises three banbury mixer models (the miget, the BR and the OOC) with respective approximate melt net mixing chamber volumes of 0.4, 1.5 and 4.3 liters, and corresponding approximate mix capacities of 0.3, 1.2, and 3 kg. Another supplier is Haake buchler is instruments Inc., USA (mixing chamber volume range 30 - 500 ml).

(b) *Specialised instrumented equipment for investigation and study of compounding behaviour and effects.*

Equipment used for these purposes, onginally a relatively simple from of instrumented torque rheometer, has today reached a high degree of sophistication and versatility. The well-known brabender Data-processing plasti-corder represent advanced modern equipment embodying these features of a computerised modular system employing a number of interchangeable attachments ('measuring heads') which enable quantitative data to be obtained on the processing and processing-related properties of plastics composition in dry (powder) mixing, liquid-state mixing, melt compounding., and extrusion. Other torque rhemoters include the RAPRA instrument, and equipment of this kind available from haake buchler instrument Inc. (USA).

The mixing of compounding expirment, equipment and record displays and records a plot (plastogram') of the terque and stock temperature as functions of processing time. The progress, ease, and duration of mixing are reflected by the shape of the terque vs time curve, and the magnitudes of the parameter value at the characteristic points. the energy input as also calculated.



Schematic representation of three measureing head of the plastici corder system

The usual procedure is as follows[4] The mixer os preheated to the required temperature (88^0C in ASTMD 2396), the PVC polymer powder is poured onto the mixing bowl, the mixer is started, and run for about 5 minute to warm the powder up. The plasticiser is then added (through a distributing funnel), whereupon the terque rises steeply as the stock becomes lummpy (the consistency has been compared with that of wet snow)[4]

Completion of the absorption of all the plasticiser into the polymer particles (the 'dry point') is marked by a sudden drop of terque The terque may then increase some what again in line with the degree of any swelling of the polymer particles by the plasticiser (dependent upon the natures of both) The time between the moment of plasticiser addition and the dry point in the mixing tune. For the same polymer plasticiser ratio at the same mixing temperature, this time is dependent on the identities of two components,as is the extent of terque rise.

Other factors being equal , the mixing time is also a function of the components' ratio. Varying the total amount of mix at the same ratio does not substantially affect the mixing time but the terque value are higher for greater quantities of mix. The scope for evaluation and comparison of the effects of the kinds polymer and plasticiser used (as well as the presence and nature of formulation components), their proportions, and the mixing temperature , is self-evident even from this brief outline of the procedure.

A fusion test, relevant to the process and general ease of plastication and fusion of a PVC composition (rigid, semi-rigid, or flexible) in melt-compounding or processing is typically run in a roller mixer attachment, often in accordance with the method of ASTM D 2538. A rapid-loading chute is normally used to introduce the composition (usually a powder blend) into the mixer as quickly as possible (within 20 s at most) for best reproducibility and comparability of test results.

As the loading proceeds, the terque vs time curve first rises very steeply to a sharp peak (the loading speak): it then drops rapidly, reflecting the decreasing resistance of the stock to shear as it is being heated and compressed, to rises more gradually again to a second, lower peak marking the 'fusion point' at which all the polymer particles are merged into a melt the time between the loading peak and the fusion point is the fusion (plasticating) time. There after the terque drops gradually, as less force is required to continue mixing and homogenising the fused stocked.

For a particular mixing temperature, the terque value at the fusion point can be related to the proper required for the fusion of a particular compositions in a given production machine.

The fusion time can similarly serve to indicate the conditions in a production machine (screw speed in relation to screw length in screw compounder. rotor speed and pressure in an internal mixer) to be used for statisfactory fusion of the composition. A full discussion of the operation and application of the bender data-Processingand be found in ref. 4.

PROCESS ENGISEERING

Variables in the preparation of blends : besides producing the PVC composition, the mixing operation is intended to impart those properties to it which will facilitate problem-free storage, conveying and processing in the particular process. the most important of these properties include:

(i) homogeneous dispersion of the individual components throughout the blend:
(ii) blend dryness, i.e. all additives, such as stabilisers, lubricants,etc, should be comminuted. homogeneously distributed and melted, and liquid additives such as plasticiser should be worked into the PVC polymer particles : any

existing moisture should also be driven off:
(iii) good free flow flowing properties:
(iv) no tendency to segregate;
(v) low moisture absorption;
(vi) increased bulk density;
(vii) good self life of final blend

Mixing tools shape and setting : Mixing tool profile have tobe designed to prevent undue overheating of the material and build-up on the mixing flights. tool profile is extremely important in high-speed mixers in conjunction with the peripheral speed, which --as already mention between 20 and 50 ms^{-1} . Because the peripheral speed determines the energy transmittable to the product.

It is an important factor to consider when selecting a mixing system. Scorching can also occure if the individual PVC particles are heated by impact against the mixing tool: as this continues, the stabilisation of the PVC may prove inadequate and flecks or streaks of scorched material will appear in the final product.

To prevent overheating. it is important that flow along the mixing tool surface should be uniform, enabling material particles lying directly in the path of a mixing tool flight to evade it and follow the flight profile without undergoing excessiving impact or friction. For these reason, the profile of an efficient mixing tool should be aerodynamic so that its flights allow the particles to flow off rapidly, and overheating of material on its surface is consistently avoided.

Power input and temperature pattern: general aspect of the process sequence : The sequence of events in a high-speed mixer can be described as follow: When the machine is started, turbulent movement of material is set up immediately and the mixing of the individual components begins.

The material is spun against the tank wall by the mixing tools and forced upwards. Thus it leaves the area of the mixing tools, following a toroidal path to the center of the tank, to the bottem and back to the mixing tool area. Whilst undergoing this horizontal--vertical motion in the direction of tool rotation, the material starts to heat up.

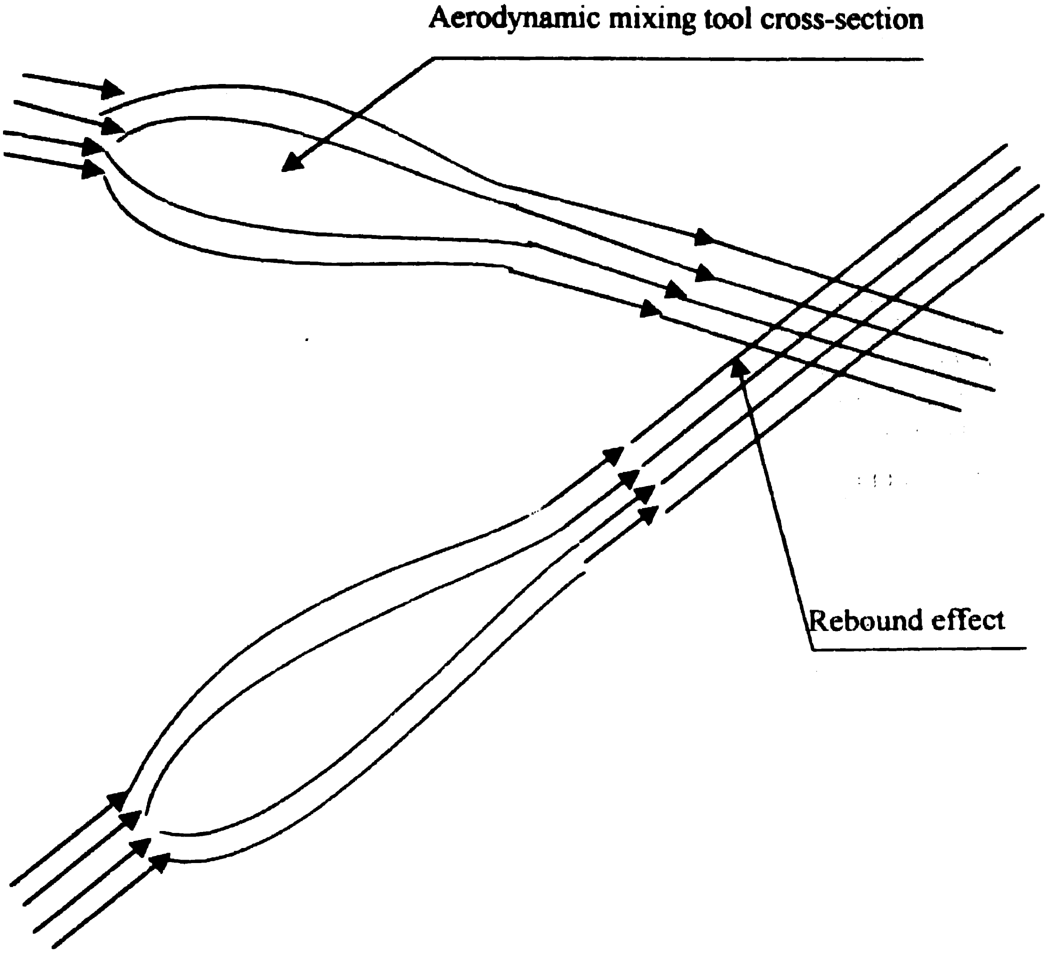
The mechanisms contributing to heat build-up are :

--- friction between material and mixing tool;
--- inter-particle friction;
---friction between air and mixing tools;
--- friction between material and tank wall;
---heat transfer from heated tank wall to air and material;
---heat generation by moving air and product;
---radiation heating

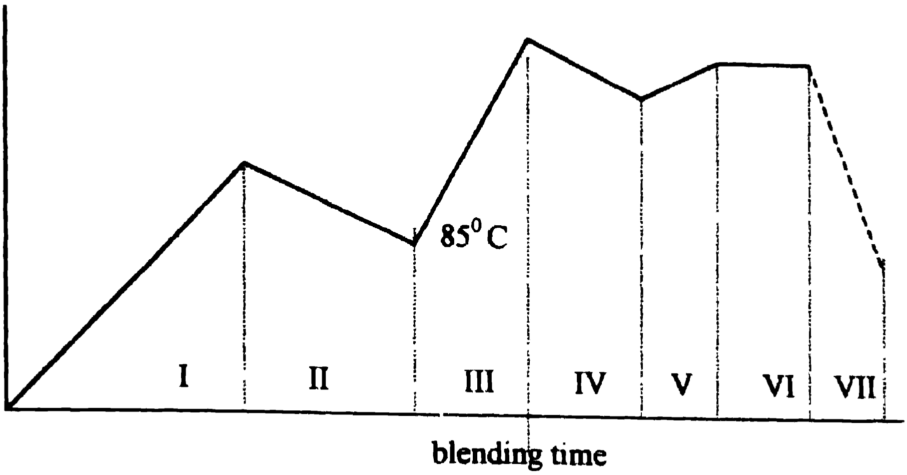
The mixing process is quasi-adiabatic, as the material is heated primarily by the mechanical energy imparted by the effect surfaces of the mixing tools and by the mutual impacts of the particles. Compared with these effects, the other heating mechanisms contribute relatively little to the heat build-up.

If the mixing tools are properly designed, external heating is required only in certain cases; for example it is advantageous where emulsion polymer is being mixed with a high percentage of plasticiser.

The best way to make mixing more gentle is to reduce the peripheral speed of mixing tools. In this case, of course, the shape and configuration of the tools must be adapted 'o the lower mixing speeds.



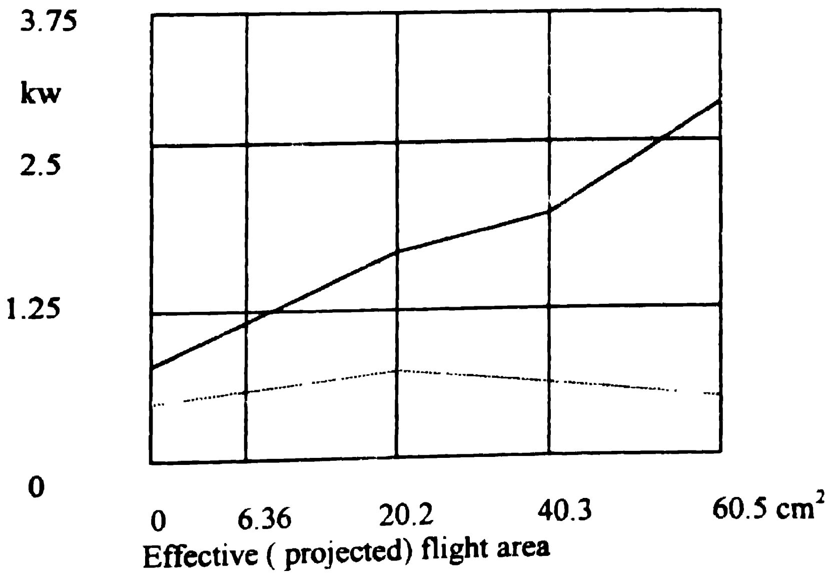
Mixing tool cross-section

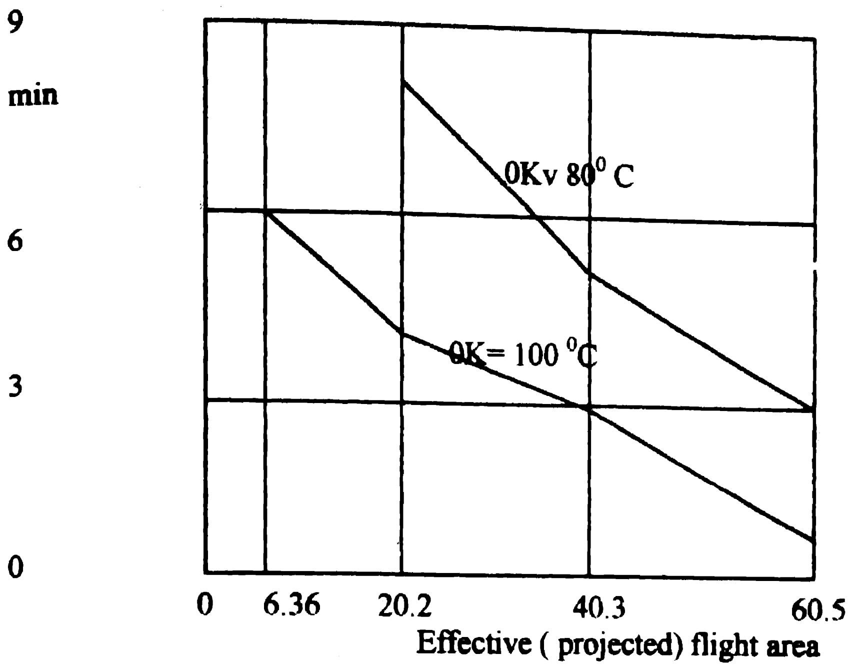


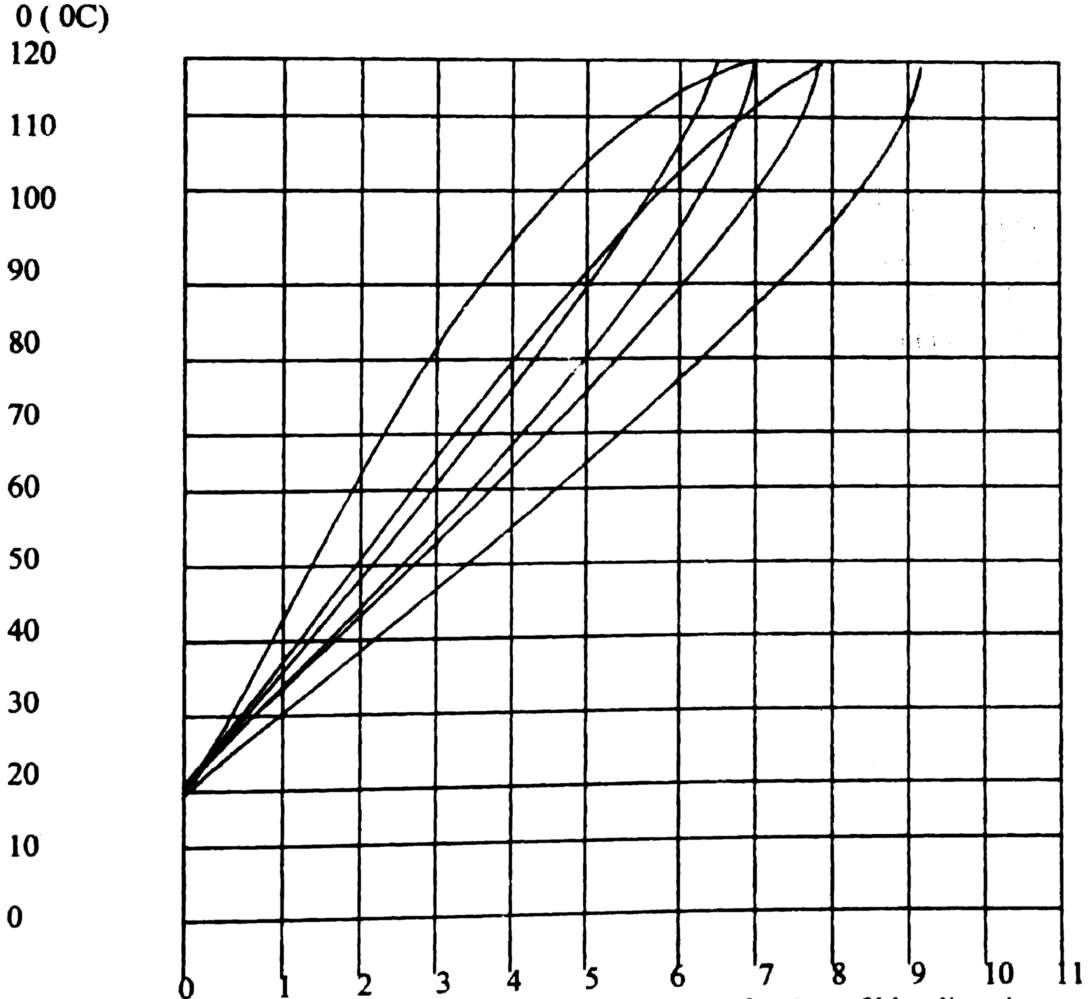
Powder input during the blending of rigid PVC in a high-speed mixer. Phase1. mixing and abarding, 2. increase in free-flowing property;3. melting of lubricants,4. dry mixing 5. melting of melting of metallie stearates:6. hot mixing.7 cooling of blend in cold mixer.

When the motor amperage levels off, the mixing operation is over and it is time to discharge the material into the cold mixer. Continued mixing in this temperature range, but below the melting point of the PVC, would merely impair the flow properties as a result of further size reduction. accompanied--perhaps surprisingly--by a drop in bulk density. Thermal degradation would also be initiated.

The energy input and temperature curves of an emulsion PVC blend with 35% plasticiser. In this case the cold plasticiser was fed continuously into the preheated mixture.



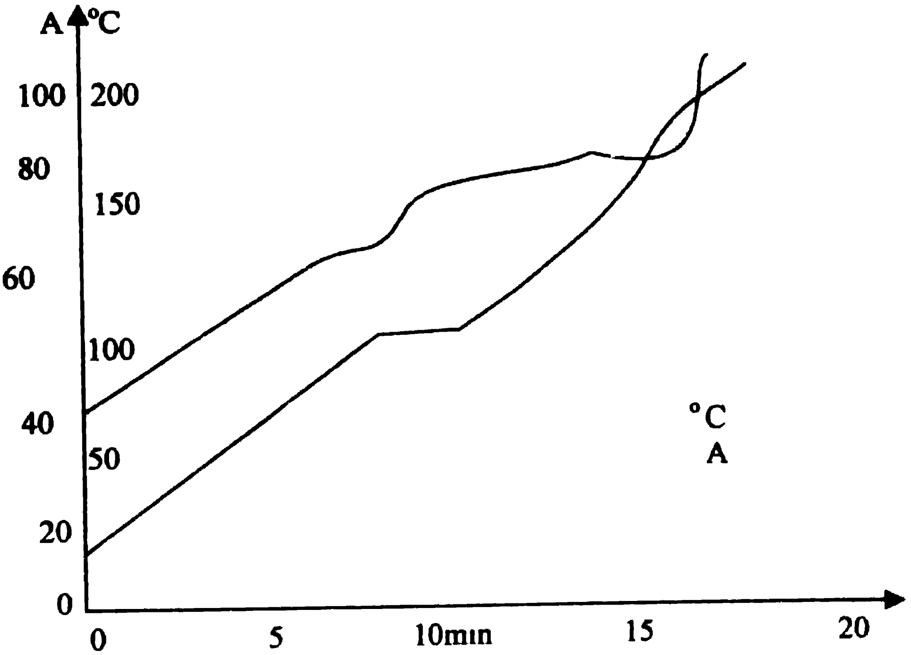


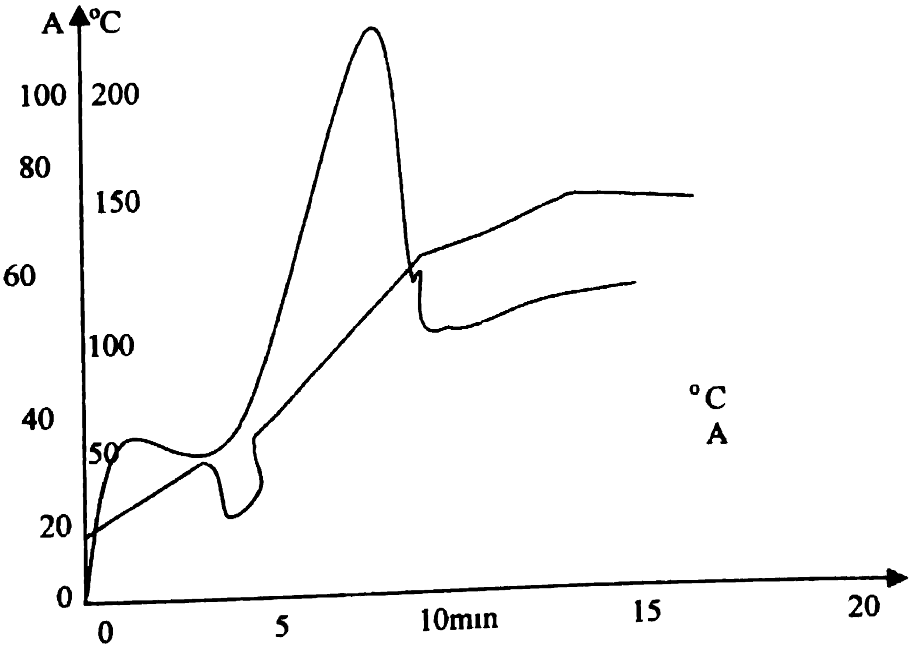


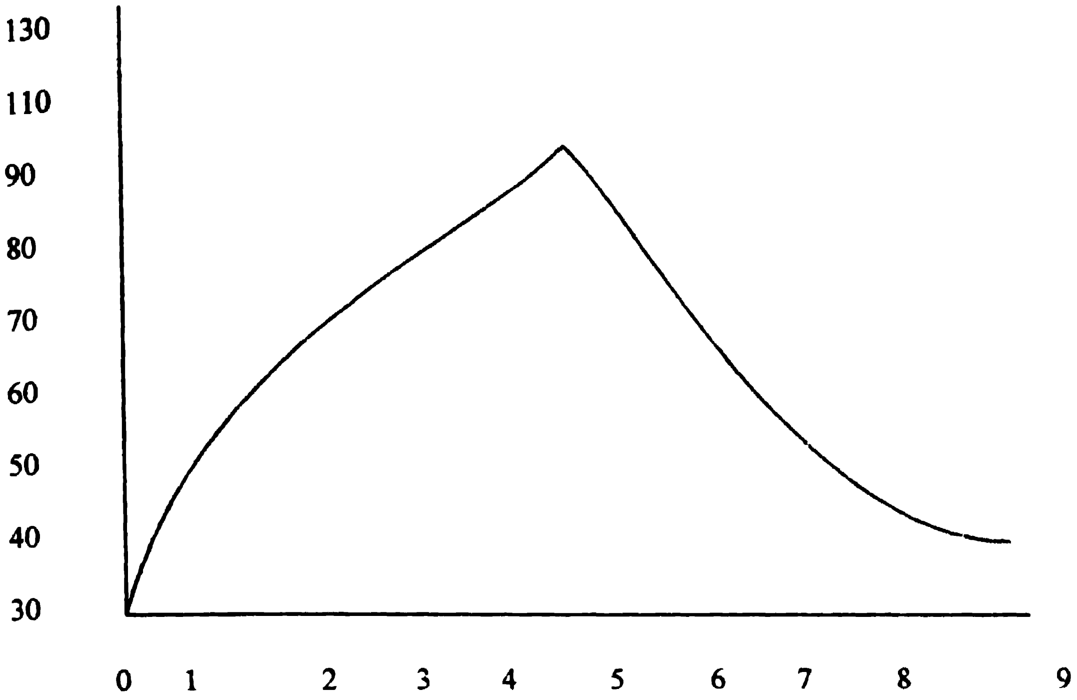
Temperature change in a high-speed mixer as a funtion of blending time

Influence of raw material components and processing conditions on the properties of hot blends:

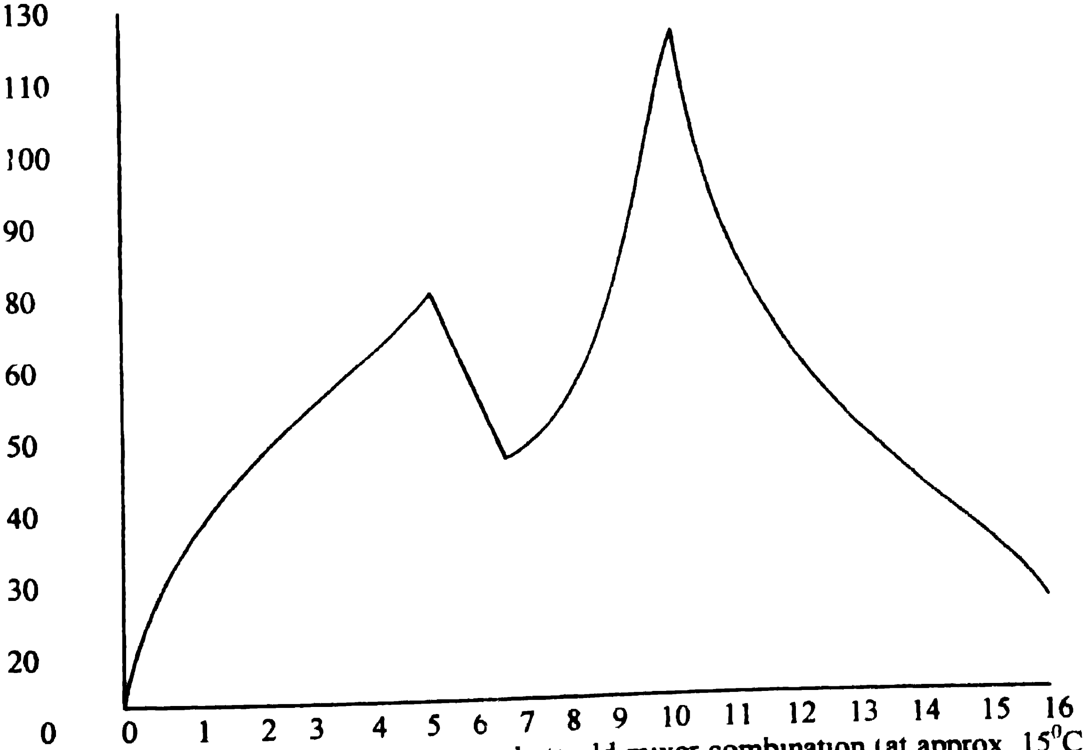
---PVC polymer. Particle structure, shape and size affects the blend flow properties. Suspension and bulk polymer with porous particles absorb the plasticiser easily. Fine-grained emulsion polymers containing emulsion polymer containing emulsifier, and their large-grained clusters which are broken down in the mixer, usually result in pasty stock when the plasticiser is added· this can only be used to produce agglomerater.







Temperature as a function of time in a hot/cold mixer combination (at approx 15^{0}C cooling water inlet temperature) a rigid PVC pipe blend, B, plasticised PVC shoe compound



Temperature as a function of time in a hot/cold mixer combination (at approx 15^{0}C cooling water inlet temperature) a rigid PVC pipe blend, B, plasticised PVC shoe compound

Temperature as a function of time in a hot/cold mixer combination.

---Type of plasticiser. Dry mixes are difficult to produce with plastericiser of limited compatibility and with slow-gelling plastericisers. Primary plasticiser with good gelation effect give the best results.

---Plasticiser quantity. This is a crucial factor in producing a hot dry blend. The maximum amount of plasticiser that the hot mixer will tolerate depends on the type and plasticiser type

---Plasticiser viscosity. This mainly influences the diffusion rate into the PVC particles. Medium to high-viscosity plasticiser therefore requir longer mixing times, unless their viscosity is reduced by heating before addition.

---Admixing the plasticiser. Understandably, it makes a difference whether the plasticiser is placed in the mixer first and the solid components then added to whether it flows into the cold or hot pre-mix of the other components, and whether its introduction into the running mixer is made in one shot, in portion, or continuously.

---Mixing temperature and mixing time. Mixing time depend upon the required final temperature. For high-speed mixers it ranges between 10 and 20 min, depending on the formulation and whether a hot or hot/cold mix is involved. A reliable rule-of-thumb is that a temperature rise of more than 15^{0}C min^{-1} should be avoided during mixing: otherwise local overheating can damage the mix.

---Lubricant: The effects of lubricants in the hot mix depend upon their physical state (sold, liquid. paste), compatibility, melting characteristics and quantity. A liquid lubricant (e.g. paraffin oil) can produce a sticky, non-flowing hot mix if it is poorly compatible with PVC and becomes concentrated on the particle surface. PVC-compatible liquid lubricant are absorbed into the particles with the plasticiser, unless the amount added exceeds the compatibility limit.

The melting range of solid lubricant should be chosen in such a way that (1) the lubricant is molten before the final mixture temperature is reached and is dispersed homogeneously throughout all of the material, and (2) the solid physical state has already been regained after the material has cooled down to the cold mixer temperature (about 40^{0}C)

---Filler: The effect of a filler on the production of hot mixer depend on its amount, particle size and plasticiser absorption. High percentage of very fine-grained filler impair the free flowing property. Fillers with porous surface usually have undesirable high plasticiser absorptions, but on the other hand, promote dry mixer. Mixer poorly flowing in the cold mixer can be improved by the addition of very small amounts of colloidal silica.

APPLICATION OF INTENSIVE MIXERS

Either high-speed mixer (hot mixer) or hot/cold combinations are used, depending on the production route followed for the final product

Hot mixer (high-speed mixers) Hot mixers are notrmally used alone ahead of pelletising machines and for direct processing of PVC powder blends The advantages of hot mixing of PVC powder in high-speed mixers are

—optimal mix quality and homogeneity

---short cycle times and hogh output rates

---very free-flowing blends,

---pneumatic conveying of dry blends or agglomerates without product segregation,

---up to 20-40% increase in bulk density as a result of sintering, thus raising the output rates of processing machine,

---low-cost and effecting lowering of the residual VC content, partly because of the high mixing temperature of more than 80°C,

---nearly complete elimination of residual moisture in the material

During mixing of suspension and bulk polymers, high friction sometimes produces an electrostatic charge in the material, whih impairs flow properties especially in batches with relatively low bulk densities One way to eleminate the charge ie to cool the mix rapidly to about 40°C in the cold mixer

Weight about 42 g of the material on a piece of paper. Fold a piece of black glazed paper with two parallel creases to from a channel 12.5 mm down the middle of the paper. Counterpoise the paper on the balance, then place it flat on the bench. Place a 500 um IS-sieve on the black glazed paper in such a way that there is a 50 mm space between the sieve and the paper. Transfer the weighed material to the sieve and with the black of the finger tips fitted with rubber finger stalls lightly rub all the powder through the sieve using short strokes. Transfer the black glazed paper with the sieved material to the balance and adjust the weight of the sieved material to 40 g. pick up the paper and form it into a chute Allow it to lie between the thumb and fingers on the hand and introduce it into the cylinder, which should be held at 45^0 to the vertical. Slip the powder gently and smoothly into the measuring cylinder as shown in fig. With the thumb and four fingers of one second lift it as far as 25 mm. Do not jerk the cylinder by knocking it against the uper stop. At the startof the next second, release the cylinder smoothly. Continue lifting and dropping until 50 complete drops have been given. Once in every 2 second a gentle twist of about 10 should be given to the cylinder As soon as 50 drops are completed, raised the cylindre to eye level and read the volume of the material.

Claculation

$$\text{Bulk density, g/ml} = \frac{40}{M}$$

Where

V= volume in ml of the sieved material

DETERMINATION OF CALCIUM CARBONATE

Method – Two method have been prescribed for this test, namely Method A and B. Method A is the classical method and method B is the complexometric method. In case of dispute Method A shall be followed

Mathod A

Outline of the method

Calcium is precipitated as its oxalate the precipitate is dissolved in dilute sulphuric acid and the solution is titrated against permanganate solution.

Reagent

Dilute hydrochloric acid – approximately 5 N

Ammonium oxalate solution – Saturated.

Dilute Ammonium hydroxide – Approximately 5 N and 1 N

Dilute Potassium permanganate solution- Approximately 0.01 N.

Dilute sulphuric acid – Approximately 5 N.

Standard potassium permangate solution – 0.1 N.

Procedure

$$\text{Bulk density, g/ml} = \frac{40}{M}$$

Weight accurately about one g of the material dried and transfer into a 250 ml beaker. Moisten throughly with a few milliliters of water and add dilute hydrochloric acid dropwise until solution is complete. Continiue adding the acid until a total of 40 ml has been added. Heat the solution and boil for about 10 minutes. Cool and transfer completely to a 200 ml graduate flask, add water to make up the volume and mix thoroughly. Pipette out 50 ml of this solution to a beaker and add 75 ml of water. Heat almost to boiling and add 15 ml of ammonium oxalate solution. To the hot solution, constantly stirred, add dilute ammonium hydroxide dropwise until the mixture is strongly alkaline. Let it stand for two hours. Decant the clear supemantant liquid through a filter paper with cold water. Wash the precipitate first with dilute ammonium hydroxide and then with cold water until the filtrate does not decolorize hot dilute potassium permanganate solution in presences of dilute sulphuric acid, heat at 60^0 C and titrate vessel and carry out the titration till the end point is reached Carry out a blank test with all reagent but without the material following the procedure for the test.

Calculation

$$\text{Calcium carbonate percent by mass} = \frac{20.02\,(V1 - V2)}{W}$$

Where

V1 = volume in ml of standard potassium permanganate solution used for the test

V2 = volume in ml of standard potassium permanganate solution used for the blank test.

N = normality of standard potassium permanganate solution

W = mass in g of the material taken for the test.

Method B-

Outline of the method

The solution of the material after adjustment of pH, is titrated with EDTA solution using murexide indicator.

INDIAN STANDARD
METHOD OF SAMPLING AND TESTS FOR PLASTICIZERS

This Indian standard was adopted by the Indian Standard Institution on 15 Sep 1980, after the draft finalized by the Raw materials for paint industry sectional committee had been approved by the Petroleum Coal and Related Products Division Council.

The method of test on plasticizers though common were so far covered in the individual specifications. In order to avoid any ambiguity and also to unify the various method of test it was decided to formulate a separate standard for the method of test and align the same with the corresponding overseas standards Accordingly after the publication of this standard, the methods of test covered in the various individual standard on plasticizers shall be withdrawn.

In reporting the result of a test or analysis made in accordance with this standard if the final value observed or calculated is to be rounded off it shall be done in accordance with IS 2-1960.

Scope

This standard prescribed the method of sampling and test for plasticizers

TERMINOLOGY

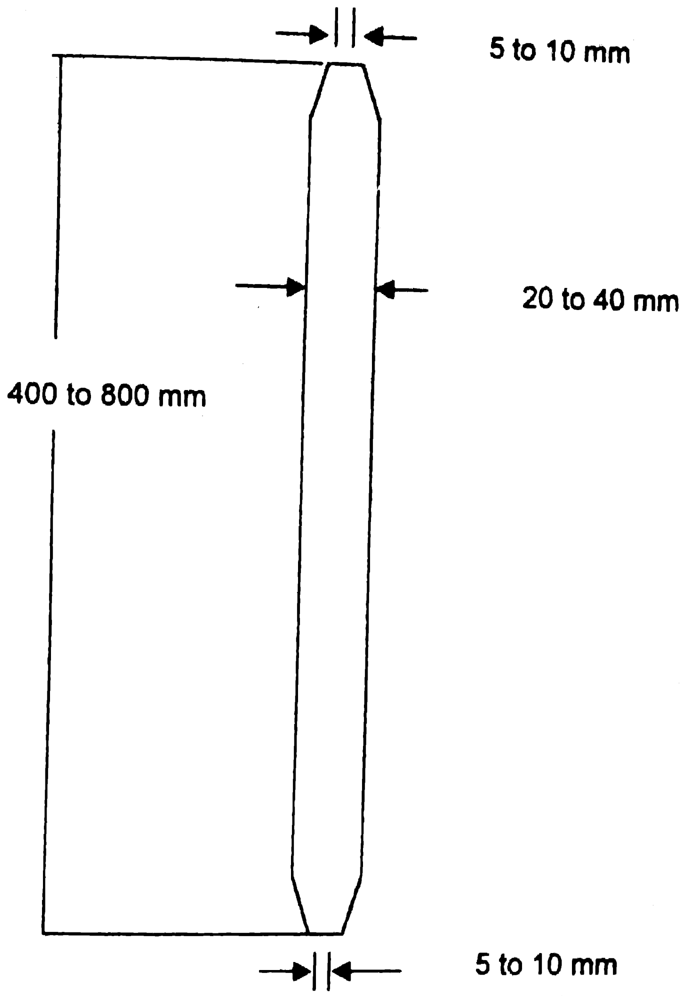
For the purpose of this standard the definition given in IS 2828-1964 shall apply

SAMPLING

General requirement of sampling

In drawing preparation storing and handling test samples the following precautions and directions shall be observed.
The sampling instrument shall be clean and dry.
The samples the materials being sampled the sampling instrument and the samples shall be protevted from adventitious contamination.
The samples shall be placed in suitable clean dry and airtight glass containers.
The sample containers shall be such a size that they are almost but not completely filled by the sample.
Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling the date of sampling and the year of manufacture of the material.
Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

Sampling instrument



Sampling tube

It shall be made of thick glass or metal on which the material has no action and shall be 20 to 40 mm in diameter and 400 to 800 mm in length. The upper and lower ends are conical and reach 5 to 10 mm diameter at the narrow ends. Handling is facilitated by two rings at the upper end. For drawing samples the apparatus is first closed at the top with the thumb or a stopper and lowered till a desired depth is reached. It is then opened for a short time to admit the material at the desired depth and finally closed and withdrawn.

For small containers the size of the sampling tube may be altered suitably

Scale of sampling

Lot – All the containers in a consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots. In the case of a consignment drawn from a continious process, 1000 containers shall constitute a lot.

The number of containers to be drawn from a lot shall depend on the size of the lot and shall be in accordance with col 1 and 2 of table.

TABLE **Number of containers to be selected for sampling**

Lot size N (1)	No. of containers to be selected n (2)
3 to 50	3
51 to 100	4
101 to 300	5
301 to 500	6
501 to 1000	7
1001 to above	8

these containers shall be selected at random from the lot. To ensure the randomness of selection a random number table as agreed to between the purchaser and the supplier shall be used. In case such a table is not available the following procedure shall not be adopted.

Starting from any container in the lot count them 1,2,3 up to r and so on in one order. Every rth container thus counted shall be withdrawn to give a sample for test where r is the integral part of N/n.

DETERMINATION OF COLOUR

Outline of the method
The colour of the samples is compared with that of permanent colour standard and expressed in terms of hazen coloue units.

Apparatus

Flat based chlorimetric Tubes- Approximately 20 mm external diameter and having a graduation mark 100 mm above the base.

Reagents

Cobaltous Chloride, Hexahydrate

Hydrochloric acid – conforming to IS 265-1976.

Chloroplatinic acid- Dissolve 1.000 g of platinum in a small quantity of aqua regia contained in a glass or porcelain basin by heating on a water bath. When the metal has dissolved evaporated the solution to dryness. Add 4 ml of hydrochloric acid and again evaporated to dryness. Repeat this operation twice more.

Potassium Chloroplatinate – 1.25 g of the salt containing 0.5 g of platinum.

Preparation of colour standards

Dissolve 2.0 g of cobaltous chloride, hexahydrate, and either the chloroplatinic acid or 2,450 g of potassium chloroplatinate in 200 ml of hydrochloric acid. Warm,if necessary to obtain a clear solution and after cooling pour in to the 2000 ml volumetric flask, Dilute with water up to the graduation mark.

From the solution prepare a series of permanent colour standard ranging from zero hazen colour unit upwards, at intervals of 10 units. For each 10 units pipette 5.0 ml of the solution into the 250 ml volumetric flask and dilute with water up to the graduation mark. Transfer each standard solution to a storage bottle. Check these standards at intervals against freshly prepared standards.

Procedure

Pour into the colorimetric tube a quantity of the sample sufficient to fill it to the graduation mark. Similarly pour the color standard selected in to the other tube to the mark. Compare the color of the sample with that of the standard by looking down the tube against the white background taking care to avoid side illumination. Repeat with other color standards if necessary until the closet match is obtained. If the color of the sample does not match that of any other of the color standards, report an approximate value for color with an appropriate note. Where matching proves impossible give a description of the color of the sample.

Expression of result

Report the result to the nearest 10 Hazen color units

DETERMINATION OF RELATIVE DENSITY

Outline of the method-

The relative density of the material is determined with a specific gravity bottle or a pyknometer.

Apparatus

Specific Gravity Bottle or pkynometer-with well fitting ground glass joints. To calibrate clean and dry the relative density bottle or pkynometer throughly weight and then fill with freshly boiled and cooled water at 27.0 +- 2^0 C after removing the cap of the side arms. Fill to over flowing by holding the bottle or pkynometer on its side in such a manner as to prevent the entrapment of air bubbles. Insert the stopper and immerse pkynometer/bottle in a water bath maintained at 27 0 +- 2^{0}C. keep the entire bulb of pkynometer/bottle completely covered with water and hold at that temperature for 30 minutes. Carefully remove an water which has exuded from the capillary opening. Remove pkynometer/bottle from the bath wipe completely dry, replace the cap bringback to 27.0 +- 0.2^{0}C and weight. Calculate the mass of water. This is a constant for the bottle or pkynometer but should be checked periodically.

Water bath- maintained at the desired test temperature.

Thermometer- any convenient thermometer of a suitable range with 0.1 or 0.2^{0}C sub-divisions. The thermometer should be checked against a standard thermometer, which has been calibrated and certified by the natinal physical laboratory, New Delhi or any other laboratory recognized for such work.

Procedure

Fill the relative density bottle or pkynometer with the material previously maintained at 27.0 +- 0.2^{0}C. hold the bottle or pkynometer on its side in such a manner as to prevent the entrapment of air bubbles after removing the cap of the side arm. Insert the stopper and immerse in the water bath maintained at 27.0 +- 0.2^{0}C for a period of at least 30 minute. Carefully wipe off any material that has come through the capillary opening. Remove the bottle the cap of the side arm, bring back to 27.0 +- 0.2^{0}C and weight.

Calculation

$$\text{Relative density at } 27/27\text{ C} = \frac{M1 - M}{M2 - M}$$

Where

M_1 = mass in g of the specific gravity bottle or pkynometer with material at the test
temperature,

M = mass in g of the specific gravity bottle or pkynometer

M2 = mass in g of the specific gravity bottle or pkynometer with water at the test
Temperature

DETERMINATION OF REFRECTIVE INDEX

Outline of the method

The refractive index is determine by abbe Refractometer at 27 +- 0 1^0 C is used. Water supply is used for maintaining this temperature

Procedure

Place the refractometer in front of a suitable source os a light. Adjust the temperature of refrector prisms with alcohol and wipe them dry. Spread a drop of liquid to be tested on the lower prism and clamp it. Adjust the mirror so that the light enters the telescope. Focus the eye piece on the cross hair and the reading lens of the scale by moving the prism arm and find a position where the lower part of the fields is dark and the upper part is bright In general the border line which is coloured shall be coloured by turning the milled head on the right of the telescope until a sharp line of contract between black and white areas is obtained.

Move the prism-arm until the black edge just cross the inter section of the cross hair. Read the refractive index on the scale to the fourth decimal place. The accuracy of the instrument should be checked by a small test piece of known refractive index which is supplied with the refractometer This test piece is attached to the upper prism with a liquid of high refractive index.

DETERMIANTION OF WATER CONTENT

Moisture is determined by the Karl fischer method
Take about 20 g of the material weight accurately and determined the water content by the procedure given in IS 2362-1973

DETERMINATION OF ASH

Procedure

Slowly burn approximately 50 g which to the nearest gram of the material in several portions in a weighted platinium or silica basin and ignite finally in a furnace at 600 +- 30°C until a carbonaceous matter has disappeared. Cool in a desiccator and weight.

Calculation

$$\text{Ash, percent by mass} = 100 \times \frac{M1}{M2}$$

Where

M_1 = mass in g of the residue

M_2 = mass in g of the material taken for the test.

DETERMINATION OF ACIDITY

Outline of the method

Acidity is determined by titration with standard solution of sodium hydroxide using phenolphthalein as indicator.

Reagent

Rectified Spirit- conforming to IS 323-1959

Standard Sodium Hydroxide Solution- 0.1 N.

Phenolphthalein Indicator- Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit and make faintly pink by the addition of dilute sodium hydroxide solution.

Procedure

Take 50 ml of rectified spirit add 0.5 ml of phenolphthalein indicator and neutralize with standard sodium hydroxide solution until the pink colour persists for 5 seconds. Add 50 g of the sample weighed to the nearest 0.5 g and titrate the mixture with standard sodium hydroxide solution until the pink colour persists for seconds.

Calculation

$$\text{Acidity percent by mass} = \frac{0.83\,V}{M}$$

Where

V = volume in ml of sodium hydroxide solution used

M = mass in g of the material taken for the test.

DETERMINATION OF ESTER VALUE

Outline of the method

The ester present is saponified by potassium hydroxide in ethanolic solution and then titrated with standard hydrochloric acid.

Reagents

Potassium Hydroxide Solution – approximately 1 N solution in rectified spirit

Standard Hydrochloric Acid- 1 N

Phenolphthalein Indicator- Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit and make faintly pink by the addition of dilute sodium hydroxide solution.

Procedure

Weight accurately about 2.5 g of the material in 10 ml conical flask fitted with ground glass stopper. Add accurately with a pipette 50 ml of potassium hydroxide solution and 5 ml of freshly refiled and cooled water. Attach the flask to water cooled reflux condenser and heat for one hour on a boiling water bath. Carry out a tank test simultaneously. After cooling wash down the inside of each condenser twice with 20 ml of freshly boiled and cooled water. Disconnect the flask and wash each joint with furthur 20 ml of water. Add 25 ml of phenolphthalein indicator and titrate the mixture immediately with standard hydrochloric acid solution until pink colour is discharged.

Calculation

$$\text{Easter value} = \frac{56.1(V2 - V1)}{M} - 6.75A$$

Where

V2 = volume in ml of standard hydrochloric acid solution required for blank

V3 = volume in ml of standard sodium thiosulphate solution required for titration

M = mass in g of the material taken for the test.

DETERMINATION OF MELTING POINT

Apparatus

Melting Point Tubes – thin walled uniform bore, capillary glass tubes open at both ends and with the following dimentions;

a) Length – 50 to 60 mm
b) Inside diameter – 0.8 to 1.1 mm

c) Outside diameter – 1.2 to 1.5 mm

Thermometer – with 0.20 subdivision and a suitable range. The thermometer should be checked against a standard thermometer which has been calibrated and certified by the National Physical Laboratory, New Delhi, or any other laboratory recognized for such work.

Heat Source – Gas burner or electric hot plate with rheostat control.

Procedure

Melt the sample and filter it through a filter paper to remove any impurities and the test traces or moisture. Make sure that the sample is absolute dry. Mix the sample throughly. Insert a clean melting tube point tube in to the melton product so that a column of the material about 10 mm long is forced in to the tube. Cautiously fuse one end of the tube in a small flame, taking care not to be burn the sample. Place the tube in a beaker and while the sample is still in the liquid state, transfer to a refrigerator and hold at 4 to 10^0 C overnight. Remove the tube from the refrigerator and attach with a rubber band or by any other suitable means to the thermometer so that the lower end of melting point tube is even with the bottom of the bulb of the thermometer. Suspend the thermometer is immersed in a large test tube containing water and immerse it in a 600 ml beaker which is about half full of water. The bottom of the thermometer is immersed in the water about 30 mm below the surface. Adjust the starting bath temperature from 8 to 10^0 C below the melting point of the sample at the begning of the test. Agitate the water in the large test tube as well as in the beaker with a small stream of air or by other means, and apply heat so as to increase the bath temperature at the rate about 0.5^0 C per minute. Heating is continiue until the liquid in the tube is completely clear throughout. Observe the temperature at rate

etermine as the melting as the melting point, provided that the readings do not iffer by more than 0.5°C.

ETERMINE THE IODINE VALUE

ut line of the method: The material is treated with a known excess of iodine onochloride solution in glacial acetic acid. The excess of iodine monochloride is etermined iodometrically.

pparatus

hermometer – An engraved stem therometer, calibreated between 10° and 65° in 0.1° intervals and with the 0° C point marked on the stem, is recommended. he thermometer shall have an auxiliary reservoir at the upper end, length of bout 370 mm and diameter of about 6 mm.

eagent

arbon Tetrachloride or Chloroform

cetic Acid – Glacial, 99 percent having a melting point of 14.8° Cand free from educing impurities. Determine the melting point.

alculation

$$\text{odine value} = \frac{12.69\,(B-S)\,N}{M}$$

Vhere

= volume in ml of standard sodium thiosulphate solution required for the blank,
= volume in ml of standard sodium thiosulphate solution required for the aterial,
= normality os standard sodium thiosulphate solution,
= mass in g of the material taken for the test.

ETERMINATION OF RESIDUE ON IGNITION

eagent

oncentrated Sulphuric acid

rocedure

Veight accurately about 10 g of the material in a tared crucible. Ignite gently at rst, until the substance is throughly charred. Cool and moisten the residue with

reagent as used with the material. Camoare the colour produced in the two Nessler cylindres.

The material shall be taken to have not exceed the limit prescribe the intensity of colour obtained with the sample is not greater than the produced in the control test.

DETERMINATION OF MOISTURE

Outline of the method- Water is removed by distilling the material with toluene and the volume of water thus isolated is noted.

Apparatus
The apparatus shown in fig consists of the following parts

a) Flask A- of 500 ml capacity made of hard resistance glass.
b) Trap B – which is 235 to 240 mm in length and the distance between connecting tube D and receiving tube E shall be about 70 mm. The internal diameter of the connecting tube is 14 mm. The cylendrical portion of the receiving tube is 95 to 120 mm in length and the internal diameter of neck of the trap is 28 mm. The receiving tube is graduate to contain 2 ml and is subdivided in to 0.1 ml division. The error in any indicated capacity should not be greater than 0.02 ml.
c) Condenser C- This is approximately 400 mm in length and the bore of the inner tube of the condenser is 8 to 10 mm. The condenser is connected to the trap as shown in the figure.

Reagent
Toulene – treated with excess of water and distilled.

Procedure
Weight accurately about 20 g of the material and transfer it in to the flask. Add about 200 ml of toluene and a few pieces of dry pumic stone. Connect the apparatus and fill the receiving end of the trap with toluene poured through the top of the condenser. Heat the flask gently for 15 minutes and when the toluene begins to boil, distil at a rate of 2 drops per second until most of the water has passed over. Then increase the rate to about 4 drops per second. When the water has apparently all distilled over, rinse the inside of the condenser tube with toluene while brushing down in the tube with a brush distillation to a copper wire brushing down in the tube with a tube brush attached to a copper wire and saturated with toluene. Continue the distillation for the 5 minute, then remove the source of heat, and allow the receiving tube to cool to room temperature. If any

Calculation

$$\text{Acid value} = \frac{5.61\,V}{M}$$

Where

V = volume in ml of standard potassium hydroxide solution used in titration and

M = mass in g of the material taken for the test.

DETERMINATION OF MONOSTEARATE AND FREER GLYCERINE CONTENT

Outline of the method

Free glycerine is separated by washing an ethylacetate solutiob of the sample with aqueous sodium sulphate solution. Glyceryl monosterate and glycerine are then determined separately by treatment with excess of periodic acid which is titrated iodometrically.

Reagents

Ethyl Acetate

Sodium Sulphate Solution – 10 percent solution of the decahydrate.

Glacial Acetic Acid

Concentrated Periodic Acid Reagent – Dissolve 11.0 g of periodic acid in 200 ml of water and add 800 ml of glacial acetic acid to it.

Potassium Iodide Solution - 25 percent solution

Standard Sodium Thiosulphate Solution – exactly 0.2 N

Starch Indicator Solution – freshly prepared 1 percent solution in water.

Procedure

Weight accurately about 0.9 to 1.1 g of sample in to a 100 ml separating funnel. Add 25 ml of ethyl acetate and dissolve the sample by heating the funnel, while well stoppered, on its side on a hot plate, shaking occasionally to facilititate solution. After the sample is completely dissolved cool the funnel and contents under tap water to cool the ethyl acetate vapours and to prevert possible loss of

solution on opening the separating funnel. Wash the solution three times with 10 ml portions of sodium sulphate solution carefully separating the lower aqueous glycerol layer each time. These washing are preserved for the estimation of free glycerine.

Transfer the ethyl acetate solution into 500 ml iodine flask. Wash the separating funnel twice with 10 ml portions of glacial acetic acid and combine these washing with the ethyl acetate solution in the iodine flask. Wash down the neck of the iodine flask with 5 ml of glacial acetic acid. Add from a pipette exactly 50 ml of concentrated periodic acid reagent of the content of the iodine flask. Stir the flask and content on a hot plate with a rotary motion for 45 to 60 seconds. Usually the solid fats will be dissolved by this time. Keep the flask for 15 minutes on the table. Add 20 ml of potassium iodine solution and titrate rapidly with standard sodium thiosulphate solution. Near the end point add a few milliliters of water and complete the titration with vigourous shaking to pure milky white colour. The end point transition is blue to yellow to white. Carry out a blank containing 25 ml of ethyl acetate and 25 ml of glacial acetic acid and the same volume of periodic acid reagent and potassium iodide solution. Allow the blank to stand for 15 minutes without heating.

Transfer the aqueous glycerine layer preserved to a 500 ml iodine flask. Add 50 ml of concentrated periodic acid reagent from a pipette to the content of the flask. Mix by swirling for several seconds. allow the contents of the flask to stand for 10 to 15 minutes at room temperature. Then proceed, use the same blank as obtained.

Calculation

$$\text{a) Monosterate content percent by mass} = \frac{3.44\,(V1 - V2)}{M}$$

$$\text{b)Free glycerine percent by mass} = \frac{0.4603\,(V1 - V2)}{M}$$

Where

V1 = volume in ml of standard sodium thiosulphate solution required for titration of
blank,

V2 = volume in ml of standard sodium thiosulphate solution required for titration of
sample

INDIAN STANDARD

SPECIFICATION FOR GLYCERYL MONOSTEARATE FOR COSMETIC INDUSTRY

This Indian standard was adopted by the Indian Standard Institution on 11 Nov 1985, after the draft finalized by the Raw materials for paint industry sectional committee had been approved by the Petroleum Coal and Related Products Division Council.

This standard was originally published in 1967 and subsequently revised in 1977. Initially while reviewing this standard the sectional Committee had agreed to indicate separately the essential and optional requirements. Subsequently this was found non implementable for certification and therefore in this revision only a single set of requirements has been stipulated.

Glyceryl monostearate is widely used in cosmetic pharmaceuticle and food industry as a blending, dispersing and stablizing agent. In the cosmetic industry it is used in preparation of hair dyes shampoos and in cold and other creams.

For the purpose of deciding wheather a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with IS :2-1960. The number of significant places retained in the rounded off value should be the same as that specified value in this standard.

DETERMINATION OF TITANIUM DIOXIDE CONTENT

ALUMINIUM REDUCTION METHOD

Outline of the method-

This method is similar to zine reduction method but easier and quicker to operate. Titanium is reduced to trivalent state by aluminum and titrated against ferric ammonium sulphate using ammonium thiocyanate as indicator.

Apparatus

Delivery Tube- made of 4 mm internal diameter glass tube bent in such a way as to have a horizontal run of about 150 mm and vertical drop of about 75 mm at one end, and a verticle drop of about 150 mm at the other end.

Long neck flask- 500 ml capasity.

Reagents

Ammonium Sulphate-

Sulphuric acid- relative density 1.84

Hydrochloric acid

Aluminium Foil – electrolytic grade.

Sodium Bicarbonate Solution – saturated.

Ammonium Thiocyanate Indicator Solution:-
Prepared by dissolving 24.5 g of ammonium thiocyanate in 80 ml of hot water filter and cool to room temperature, dilute to 100 ml and preserve in a well stoppered amber coloured bottle.

Standard Ferric Ammonium Sulphate Solution- Prepare by dissolving 30.16 g of fresh ferric ammonium sulphate in 800 ml of water containing 15 ml of concentrated sulphuric acid. Add 0.1 N potassium permanganate solution until a very slight pink colour is obtained. Dilute to 1000 ml and mix thoroughly Filter if necessary. Standardize with 0.190 to 0.210 g of standard titanium dioxide as prescribed and calculated titanium dioxide equivalent of solution in g of titanium dioxide per ml of solution as given below;

$$\text{Titanium dioxide equivalent} = \frac{M \times P}{V \times 100}$$

Where
M = mass in g of standard titanium dioxide taken for test,

P = percent of TiO_2 in standard titanium dioxide used and

V = volume in ml of ferric ammonium sulphate solution required for titration.

Procedure

Weight accurately 0.190 to 0.210 g of dry material and transfer the same to a 500-ml long necked flask. Add 7 to 9 g of ammonium sulphate and 20 ml of sulphuric acid. Mix carefully. Heat on a hot plate to fuming and over a strong flame until dissolution is complete. Cool and add 120 ml of water and 20 ml of hydrochloric acid. Boil the contents and remove from heat.

Insert the short end of the delivery tube in to one hole of a two hole rubber stopper suitable for the bottom end in the other hole in such a way that the bottom in to the flask. Attach 1 g of aluminum foil to the bottom end of the rod by coiling and delivery tube into the flask in such a way that the foil is near the bottom of the flask and at the same time the long end of the delivery tube is near the bottom of a 250 ml beaker containing 150 ml of sodium bicarbonate solution.

Heat the flask as soon as the dissolution of aluminium is completed to gentle boiling for 3 to 5 minutes without distributing the assembly Cool to 600 C by partial immersion in water and siphon the sodium bicarbonate solution into the flask giving an atmosphere of carbon dioxide over the reduced titanium solution. Withdraw the stopper rinse the glass water catching the rinsings in the flask. Add 2 ml of ammonium thiocyanate indicator solution and titrate immediately against standard ferric alum solution to a straw coloured end point.

Calculation :

Claculate the percentage of titanium dioxide as follows

$$\text{TiO2 content percent by mass} = \frac{V \times E \times 100}{M}$$

Where

V = volume in ml of ferric ammonium sulphate solution used in the test

E = titanium dioxide equivalent of ferric ammonium sulphate solution in g/ml, and

M = mass in g of material taken for the test.

DETERMINATION OF RESISTANCE TO CHALKING

GENERAL

Outline of the method-

The pigment under test and the approved sample are converted in to paints applied to separate test panels and subjected to outdoor exposure. A piece of suitable cloth and the surface examined for any chalking.

Preparation of the paint

Paints of the sample under test and approved sample shall be prepared as given

Preparation of the test panel

Apply the two paints vertically side by side on the same panel which shall be of wood or of any other material agreed to between the purchaser and the supplier and give identical outside exposed shall be not less than 120 cm2. Determine the time taken for chalking to commence by fortnight examination of the panel by the procedure prescribed.

Procedure

Draw a piece of black cloth such as velvet silesion Italian or some other suitable cloth of approximately 25 * 100 mm size, under a load of 100 g applied through a disc of soft rubber of 25 mm diameter and about 3.2 mm thick over the surface of the paint for a distance of 50 mm in approximately one second while the panel rests in a horizontal position.

METHODS OF TEST FOR CALCIUM CARBONATE, PRECIPITATED, FOR COSMETIC INDUSTRY

QUAKITY OF REAGENTS

Unless specified otherwise pure cemical and distilled water shall be employed in tests.

DETERMINATION OF FINENESS

Apparatus

Gallie Porritt Sieve- The apparatus consists of a metal funnel terminating at the foot in a short cylindrical outlet, in which is inserted a shallow removable cup. To the bottom of the cup, wire gauze of 25-mm diameter is soldered. Water under pressure of 1.5 to 3.5 kg/cm^2 is supplied by a tube fitted with a nozzel designed to discharge a spreading jet throughout the wire gauge and the tube is so arranged that the distance of the orifice in relation to the wire guaze can be adjusted. The tube is provided with a filter to ensure removal of any solid particles from the water. A second tube which is also provided with a filter is improvised to supply more water to the funnel during the progress of the test.

The area of the wire gauze shall be sufficiently large to prevent undue loss of pressure head in the case of the first tube.

Procedure

Carry out the test separatly using 150um IS-sieve and 75 um IS-sieve.
Weight accurately 50 +- 1 g of the material. Half fill the funnel with water from the subsidiary supply, then bring the central jet into operation. Transfer the weighed

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